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Monitoring Techniques for Carbon Fiber Emissions
Evaluation B

Arthur D. Little, Inc.
Cambridge, MA

Prepared for

Environmental Sciences Research Lab.
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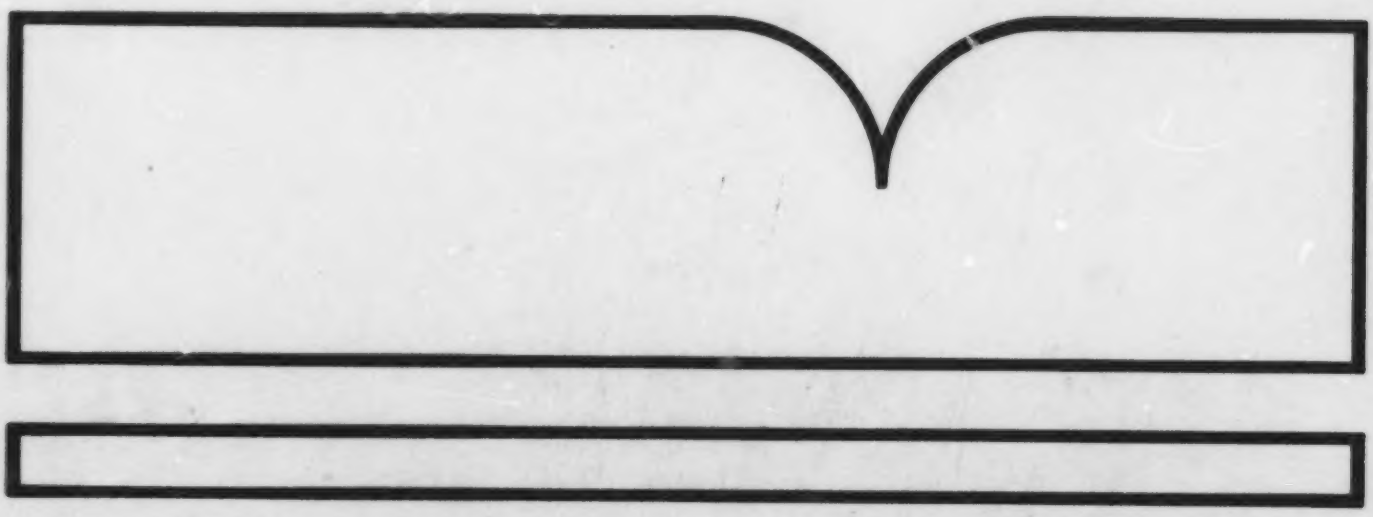

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MONITORING TECHNIQUES FOR CARBON FIBER EMISSIONS
Evaluation B

by

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Contract No. 68-02-3275

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16. ABSTRACT

An evaluation of available measurement methods for continuously monitoring the number and mass of carbon fibers emitted from source operations was conducted. A total of eleven candidate monitoring methods were identified based on contact (electrical), locally sensing (optical, microwave) and remote sensing (microwave, radar). Each method was rated on the basis of measurement (sampling), detection and instrumental parameters, and their useful concentration and length range were compared to three emission scenarios representing textile weaving, machining and waste incineration. Five methods have merit for certain conditions and are recommended for further study: for moderate to high concentrations and lengths >1 mm. microwave-OSGEF and electrical grid-arc methods; for moderate to high concentrations and lengths <1 mm, optical scattering-rotating lens and fiber aerosol monitor (FAM) methods; and for very high concentrations in absence of other particulate matter (i.e., process upset), the optical-LED method. Microwave-OSGEF is the only method that is specific to carbon fibers alone. The electric grid-arc method can be arranged to sample a major portion of the air stream, providing representative sampling. These two methods are recommended as having highest priority for further development.

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ABSTRACT

Carbon fibers released from manufacturing, application and waste disposal operations are light in weight and can be dispersed over wide areas. Because of their high electrical conductivity, they can damage electronic apparatus and electrical equipment if they come in contact. The impact of respirable fibers on health is not known. The EPA has the responsibility to evaluate and develop instrumentation for continuously monitoring the number and mass of carbon fibers emitted from source operations. The current program was conducted to evaluate available measurement methods in light of source emission characteristics.

Carbon fibers released during manufacturing and application are generally well controlled by exhaust hoods and filters. Major emission points include tow rewind, chopping, textile weaving and machining. A substantial range of fiber concentration and length distribution exist, frequently in the presence of other particulate matter, including other types of fibers.

A total of eleven candidate monitoring methods were identified based on contact (electrical), locally sensing (optical, microwave) and remote sensing (microwave, radar). Each method was rated on the basis of measurement (sampling), detection and instrumental parameters, and their useful concentration and length range were compared to three emission scenarios representing textile weaving, machining and waste incineration. Five methods have merit for certain conditions and are recommended for further study: for moderate to high concentrations and lengths >1 mm, microwave-OSGEF and electrical grid-arc methods; for moderate to high concentrations and lengths <1 mm, optical scattering-rotating lens and fiber aerosol monitor (FAM) methods; and for very high concentrations in absence of other particulate matter (i.e., process upset), the optical-LED method. Microwave-OSGEF is the only method that is specific to carbon fibers alone. The electric grid-arc method can be arranged to sample a major portion of the air stream, providing representative sampling. These two methods are recommended as having highest priority for further development.

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SECTION 1

INTRODUCTION

Carbon fiber-epoxy composites exhibit excellent stiffness and a high strength to weight ratio. These composites have been used during the past decade as a structural reinforcement material first by the aerospace industry and more recently, for sports and recreational equipment. Forecasters (1) have indicated a 25 to 40 percent annual growth over the next decade and, depending on the type of use and level of application in automobiles, could be considerably more. For several years it has been recognized that the release of carbon fibers into the atmosphere can damage electronic and electrical equipment located near the point of release. This is due to the high electrical conductivity of the fibers and the small dimensions and light weight of individual fibers, which permit their dispersion over fairly long distances. The relatively few manufacturers of carbon fibers and composite materials are aware of these problems and have taken precautions to prevent their release during manufacturing by imposing in-plant air pollution control procedures and disposing of waste materials in landfill. Nevertheless, there have been accidental releases to the atmosphere. (2)

The effects of carbon fibers have been considered by a U.S. Government interagency task force (3), with the EPA having the responsibility, among other things, to evaluate and develop instrumentation for continuously monitoring the number and mass of carbon fibers emitted from source operations such as the manufacture, processing and disposal of carbon fibers or fiber containing material. The first phase of work which is reported here relates to the evaluation of currently available instrumentation for measuring the number and mass of carbon fibers in terms of applicability to stacks or ducting to stacks (i.e., confined emissions) and performance capability (accuracy, range, reproducibility, response time, sensitivity, specificity, stability). Based on this evaluation and on the results of preliminary laboratory studies of a novel detection concept proposed by Edward Cook of Arthur D. Little, Inc., recommendations are made to the EPA in terms of the more promising systems, including the need for modifications and new development efforts.

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SECTION 2

CONCLUSIONS

There are relatively few opportunities for the release of carbon fibers during manufacturing and application, with major points of release being associated with fiber chopping, tow rewinding and textile weaving during manufacturing and with final shaping during application, including grinding, sawing and drilling. Local emissions are controlled by exhaust hoods and water sprays. Laboratory simulations of finishing operations show that the majority of fibers released are less than 0.1 mm in length. Three emission scenarios were developed to represent the range of conditions that may be encountered in the manufacturing, application and disposal of carbon fibers. These were textile weaving, carbon fiber composite machining and incineration.

Eleven measurement methods or concepts were identified as candidates for the continuous measurement of carbon fiber emissions. These candidates were scored according to parameters concerned with measurement (sampling), detection, instrument parameters and specific disadvantages, with detectable ranges of fiber concentration and length compared to the three emission scenarios. No single monitoring instrument is suitable for all possible types of emission. Fiber length and concentration ranges must be specified to permit selection of the appropriate instrument.

Monitor methods recommended for further study are:

- Moderate to high concentration, length >1 mm
  - Microwave OSGEF (Arthur D. Little, Inc.).
  - Electric grid-arc (Bionetics, JPL).
- Moderate to high concentration, length <1 mm
  - Optical Scattering-rotating lens (Epsilon Laboratories, Inc.).
  - Optical Scattering-FAM (GCA, Inc.).
- Very high concentration, only carbon fibers
  - Optical-LED (commercially available).

Microwave OSGEF is the only method that is specific to carbon fibers alone. The optical methods detect all fibers, including glass and polymer fibers. The electric grid-arc method measures all conductive fibers (and particles which cross several electrodes) and may be adversely affected by moisture content in the air stream.

SECTION 3

RECOMMENDATIONS

Up to five instrumental methods have been selected as promising carbon fiber monitors. Before a detailed development program can be recommended, it is necessary for the EPA to determine the operating parameters and conditions of interest, as no single instrument is suitable for all emission scenarios. Therefore, it is recommended that performance guidelines be developed by the EPA to permit the selection of the instrument or instruments that most nearly comply.

Based on available information, the five methods recommended for further study are

- Microwave-OSGEF
- Electric grid-arc
- Optical scattering - rotating lens (concept)
- Optical scattering - FAM
- Optical - LED

The prioritizing of developmental effort should be determined from the combined results of all EPA funded programs relating to carbon fiber emission sources, characteristics and measurement.

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SECTION 4

PROGRAM PLAN

The objective of this study was to carry out an evaluation of currently available instruments for monitoring carbon fiber emissions. In this context, carbon fibers are defined as having at least a 5 to 1 length to diameter ratio. Manufactured carbon fibers are taken to be $\geq$5 μm in diameter, whereas incompletely incinerated carbon fibers can have diameters <5 μm. The program requirements are as follows:

- The evaluation of instruments must determine their applicability to stacks or ducting to stacks (i.e., confined emissions) and performance capability (confined source sampling and analysis) for the continuous measurement of mass and number of carbon fibers in the confined emissions from source operations that include the manufacture, processing, and disposal of such fibers or fiber-containing material.

- Evaluation of performance capability shall address the following operating parameters: accuracy, range, reproducibility, response time, sensitivity, specificity, stability. Test procedures to examine these parameters shall be presented in the PHASE I report.

- A work plan shall be prepared and delivered to the EPA describing the monitoring system recommended to meet EPA monitoring needs. The work plan shall include whatever modifications and new development efforts are needed during PHASE II to provide an optimized prototype system for field evaluation. The work plan will also contain an estimate of the cost to complete PHASE II.

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The approach employed to establish the required information base was as follows:

- Develop data base on carbon fiber manufacturing and application with consideration to
  - Unit processes
  - Carbon fiber emission points
  - Control methods
  - Available sampling data

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- From this data base, describe the characteristics of typical emission scenarios for carbon fiber manufacturing, use and disposal.

- Identify instrumental methods potentially useful for monitoring carbon fiber emissions on basis of literature review and interviews with government agencies and contractors.

- Rank candidate monitor methods on basis of performance criteria according to the selected emission scenarios.

- Based on the above, recommend modifications and new development efforts required for a monitoring method to meet the EPA needs.

Initially, the effort was directed toward evaluating presently available instrumentation. Based upon certain shortcomings with all candidate monitor instruments, an additional task was added to the program to carry out laboratory evaluation of an Arthur D. Little, Inc. monitoring concept based on an optical signal resulting from interaction of carbon fibers with a high frequency electric field, referred to herein as the OSGEF method.

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SECTION 5

MANUFACTURE OF CARBON FIBER COMPOSITES

PROCESS DESCRIPTION

Carbon Fiber Manufacture

Carbon fibers (CF) are made from a number of different precursors such as resins, hydrocarbon pitches, lignin pitches, rayon, acrylic polymers, etc. Regardless of the precursor used, the processing to carbon fibers involves a series of heat treating steps to temperatures which for some fibers may reach 3000°C. A process flow sheet is given in Figure 1.

For purposes of discussion, the manufacture of carbon fibers using polyacrylonitrile (PAN) as a precursor will be used as a model (PAN is presently the most commonly used precursor, e.g., nearly 90%). This type of precursor may be purchased as either yarn, roving or tows.

The manufacture of carbon fiber may be sub-divided into three distinct processing steps, each having a characteristic temperature processing range. It should be noted that fiber may be sold, or used, as is, after any one of these three processing steps. The first step, called stabilization or oxidation, yields a non-conductive black fiber having a strength of about 50,000 psi and a modulus of about 1.5×10^6 psi. The second step, carbonization, yields a conductive fiber with a 400,000 psi tensile strength and 32×10^6 psi modulus. The third step, graphitization, provides the most conductive form and has the highest modulus, $>50 \times 10^6$ but is of somewhat lower strength, 320,000 psi.

Stabilization/Oxidation--

Typically a U.S. carbon fiber manufacturer purchases the appropriate precursor tow (based upon their technology) from Dow, duPont, Monsanto, Celanese or other textile manufacturer. These tows may differ among the various CF manufacturers in regards to number of filaments per tow, in modifier content, and in denier. A tow may contain from 1000 to 160,000 filaments (with projections up to 320,000 filaments). Modifier content may vary from 6-1% or conversely PAN content may be from 94-99%. Denier is the weight for a given length of tow; hence, in part, it relates to the filament diameter.

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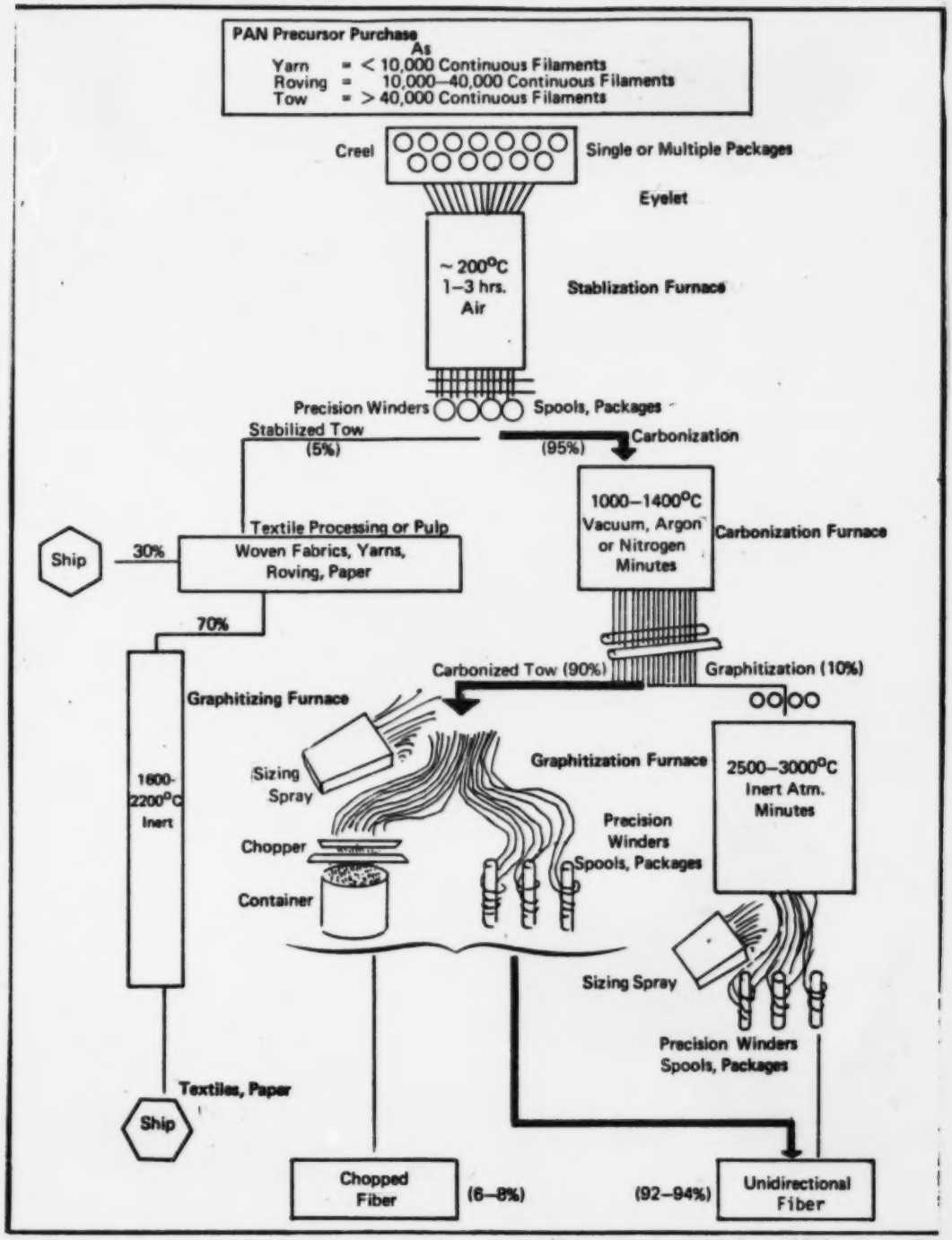

FIGURE 1 CARBON FIBER MANUFACTURING FLOWSHEET

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Subsequent to the formation of the PAN material by the textile manufacturer, it is stretched to about 10-15 times its length so as to draw out and align the main polymer chain and increase interchain adhesion. The commonly known or recognized PAN based textiles are ACRILAN, CRESLAN, ORLAN and ZEFRAN. Not all PAN based textiles are suitable for conversion to carbon fiber. The cost of these textile tows varies between $1-6 per pound depending upon denier and modifier content. Also, it should be noted that it requires about 3 pounds of precursor to produce one pound of carbon fiber.

The CF manufacturer processes either a single tow or may process a number of tows simultaneously in the form of a tape several inches in width, i.e., 4 inches. The tows are heated to about 200°C for about 2 hours in air. This oxidation-heat treatment converts the polyacrylonitrile from a linear structure to a ladder structure. The formation of this ladder renders the polymer thermally stable and prevents melting during the subsequent, higher temperature carbonization step. During this stabilization phase of the process, the filaments have a tendency to revert to their weak, unstretched state; therefore it is necessary to maintain these filaments under tension to prevent shrinkage while being subjected to the 200°C environment. After about 2 hours the filaments are quite rigid and display no tendency to shrink. This is due to the oxidation of some CH_2 groups, coupled with the loss of water and some volatile organics. The fibers at this point are nonconductive, contain about 62% carbon and have a high strain to failure ~15%.

During this stage the filament has actually undergone a weight increase due to the addition of oxygen, with essentially no change in diameter, and with a color change from white to black. This oxygen addition amounts to about 10-11% by weight.

Some fiber (about 5%) is sold in this oxidized/stabilized form as continuous tow, or as yarn, rovings or fabrics, but this material is not considered as part of the reported U.S. carbon fiber sales.

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Carbonization--

Carbonization is conducted at temperatures between 1000-1400°C in an inert atmosphere or vacuum. Little if any stretching is imposed (<2%), depending upon end use and the degree of further higher temperature processing. Upon completion of carbonization, the fibers become conductive and the carbon content has been increased to nearly 95%, with a corresponding weight loss of about 50%. The strain to failure ratio has decreased to about 1.25%. About 90% of the carbon fiber sold in the United States is sold in this form, the largest usage being in the Aerospace and Sporting Goods markets. Of this amount, 92-94% is sold as continuous fiber and the remainder as chopped-sized fiber. About 1% of the continuous fiber is chopped by the prepregger, coated and used in compression molding.

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Graphitization--

About 10% of the CF sold in the United States is graphitized material. It is more conductive, more brittle, with a higher modulus but of lower strength than the carbonized material and contains about 99% carbon. Graphitization temperatures approach 3000°C. The process is accomplished in an inert atmosphere in a matter of minutes. At this point, the fiber diameter is about 8 microns.

The subsequent, associated processing steps for preparing CF composite materials are shown in Figures 2 and 3.

Prepreg/Pultrusion

Most carbon fiber is sold as coated or sized unidirectional continuous tow and is usually processed into a more usable form by preimpregnation with a resin. The term "prepreg" is used to denote this step of the manufacturing process and consists essentially of a ready-to-mold combination of resin and carbon fiber reinforcement. Available forms of prepreg include resin impregnated woven fabrics, non-woven matt, roving and paper. These in turn can be supplied as full width rolls, tapes and sheets or as chopped material. In addition to selling unidirectional tow, sized and chopped carbon fiber is also sold.

Prepregging of unidirectional tow is accomplished by one of two techniques-solution coating or hot melt treatment (HMT). There are two essential differences between these two techniques. Solution coating uses a thermoset epoxy resin whereas the HMT uses a thermoplastic epoxy. In solution coat, the resin is dissolved in a solvent such as methyl ethyl ketone (MEK) and is applied by passing the fibers through this bath. In the HMT, the fibers pass between heated rolls which "work" the resin into the fiber.

The solution coating technique produces a product which is about 50% resin and 50% fiber by weight. This material is chopped into rod-like pellets about 1/2 inch long and 1/16 inch diameter which is then used as a feed for compression or matched die molding, the fibers being as long as the pellets.

The HMT is used to produce tape, sheet or full width (5 feet) rolls. This prepreg sheet material is packed between polyinterliners and forms the feed stock for autoclave or pressure bag rolling.

A prepreg using chopped fiber is also made by the prepregger. This material forms the feed stock for injection molded parts. In this process, a chopped fiber with sizing (a high polymeric type epoxy or thermoplastic) is blended with the pellets of an appropriate resin such as Nylon 66 or a polypherolylene sulfide. The resin contents are in the order of 80%. This mixture is then hot extruded into about 1/16 inch diameter rods, then chopped to length, about 3/16 inch. Unlike the solution coated-chopped material in which the fibers are of the same length as the pellets, the fibers in these pellets do not traverse the total length of the pellet.

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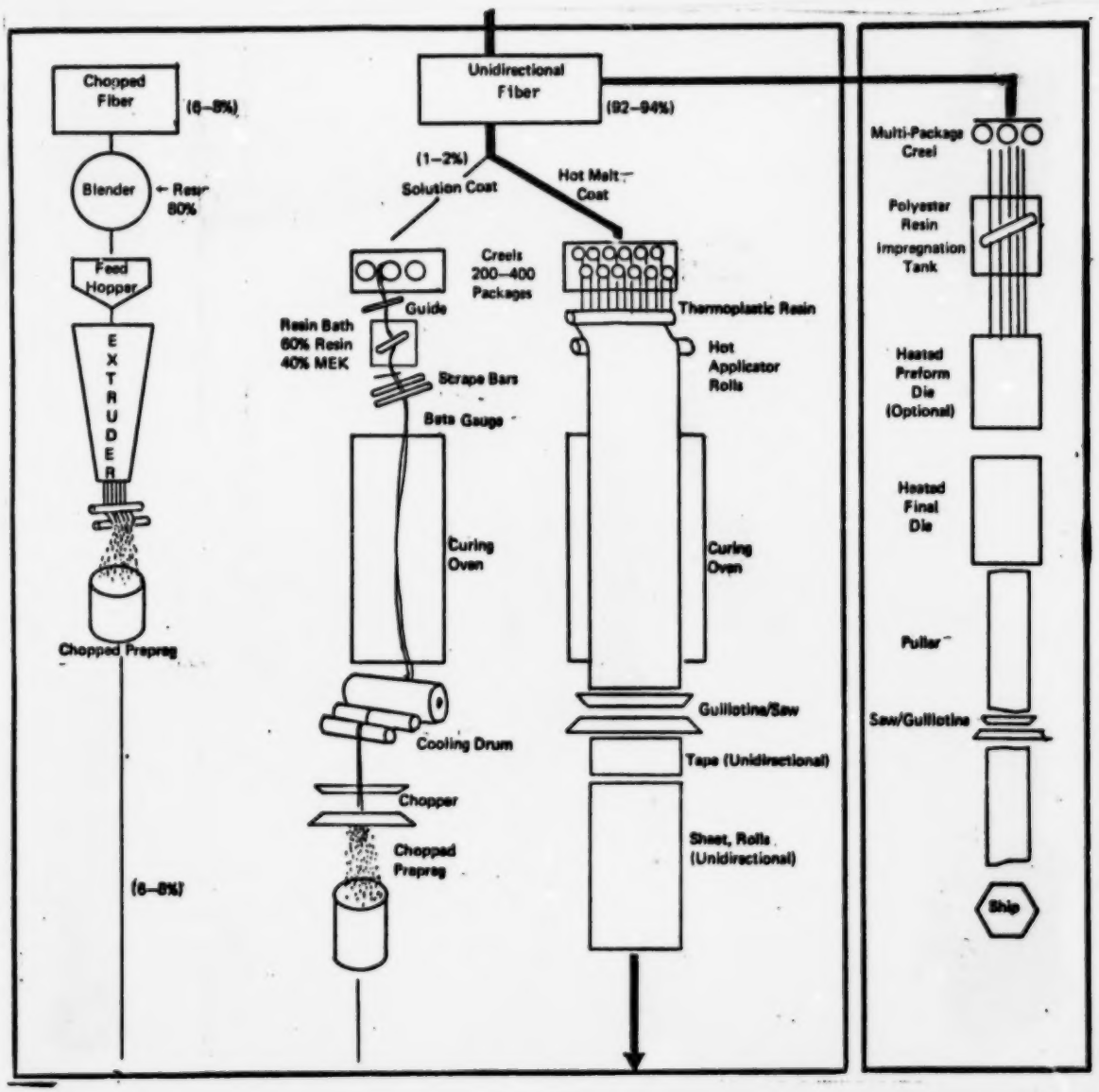

PREPREG PREPARATION PULTRUSION

Figure 2. Flowsheets for Prepreg Preparation and Pultrusion

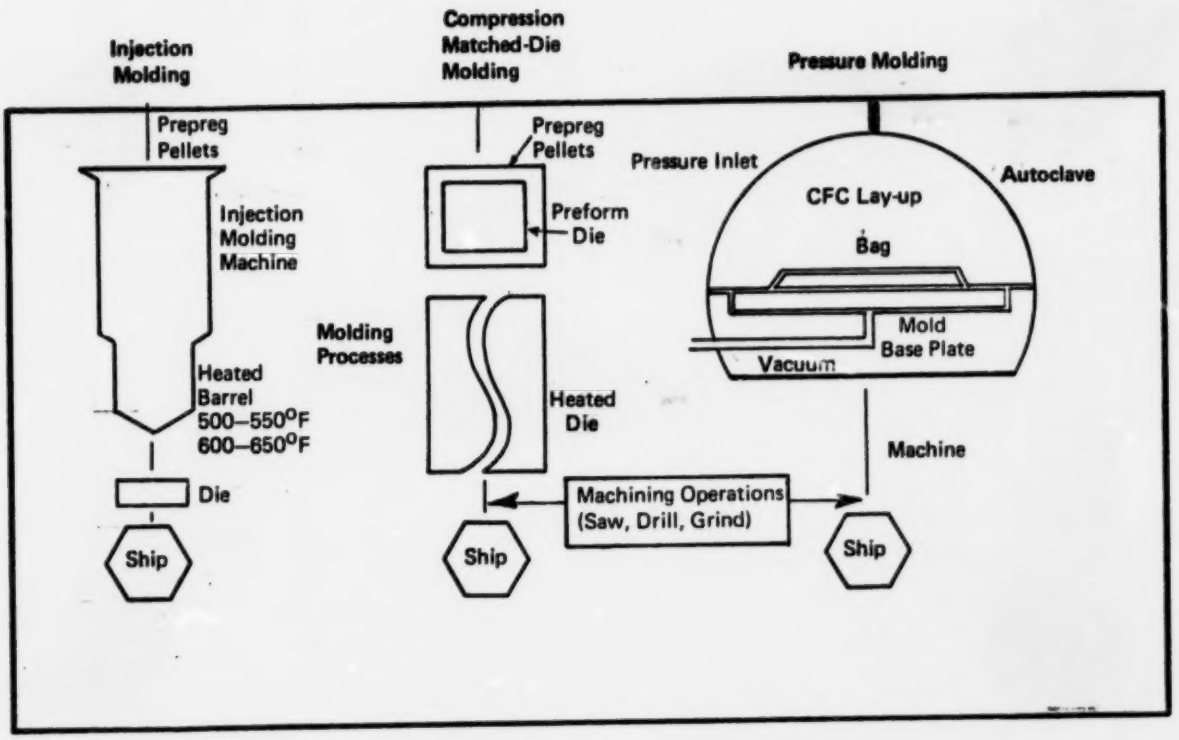

Figure 3. Carbon Fiber Composite Molding Processes

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Unlike prepreg material which must then be subsequently formed to shape, a technique which coats and forms carbon fiber in a single production line is also used. This technique called Pultrusion is shown schematically in Figure 2. Basically it consists of passing a number of tows through an impregnation tank containing a resin such as polyester in liquid form. The number of tows is dependent upon the area of the part to be formed and the loading desired. The coated fiber is then passed through a heated die at about 2 feet per minute where it is cured. Upon exiting from the die it is then cut to length, typically by sawing. For parts having a small cross section and high fiber loading, e.g., 70%, two heated dies are placed in series, the first die (preform die) being an oversized version of the second. Pultrusion is used to produce shapes of constant cross section such as bars, rods, I-beams, etc.

Forming Operations

After prepreg, the carbon fiber-B stage cured resin is then formed to shape, cured and machined. As eluded to above, prepreg material is typically molded to shape by either injection molding, compression molding or pressure bag molding, although other fiber reinforced plastic forming techniques are also possible.

Compression or Matched Die Molding--

In matched die or compression molding, both high and low pressure molding is possible. This process is ideal for long production runs and accurate control of dimensions, density and rate of cure. It consists of taking the prepreg pellets, forming a preform block and then putting these preforms into a heated die where the resin is final cured. In principle, match die molding is the bringing together of two contoured halves of a mold to impart shape. This type of molding uses solution coated prepreg as a seed material.

Bag Molding--

Typically, autoclave-pressure bag molding of CF-prepreg is used, although vacuum bag molding is also possible. Basically this is a hand layup technique of sheets of unidirectional prepreg over a mold, over which a flexible bag is placed and even pressure is applied. This setup is placed in a heated autoclave. Typical autoclave conditions are 175°C-100 psi with cure times of 2 to 8 hours. This type of molding uses layups of unidirectional-flat prepreg as a feed stock.

Injection Molding--

Basically this technique consists of taking prepreg chopped pellets, feeding them into an injection molding machine which then "squirts" the softened resin-fiber composite into an appropriate die cavity where the composite is cooled and rigidized. The part is ejected and then usually goes to a grinding operation for flash removal. This technique is used for forming intricate parts.

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Finishing Operations

After forming, finishing operations may be required to remove flashing or to produce final shape. These operations include slitting, sawing, drilling, grinding and sanding.

EMISSION CONTROL METHODS

Major emission points in carbon fiber manufacturing are associated with rewind operations (after carbonizing or graphitizing), chopping, and the weaving of carbonized material. The first two processes are controlled by local hood exhausts which exit to bag filters. These are either individual units (like an industrial vacuum cleaner) or are distributed to a central exhaust duct. Carbon fiber weaving is carried out in isolated rooms, with exhaust air vented through a HEPA room filter. Manufacturing space is run at a slightly negative pressure to prevent fugitive emissions. Another possible point of emissions is the off-gases of the carbonizing (or graphitizing) furnace, which is operated at industrial vacuum levels. These off gases are passed through an incinerator (or scrubber) to destroy noxious components and to oxidize pyrolysis products. Electrical switches and junction boxes are carefully sealed or even purged with an inert gas flow to prevent short circuits. As carbon fibers act as a skin irritant (like fiber glass), most plants are kept as clean as possible to avoid worker discomfort as well as to maintain production capacity. Carbon fiber emissions from a manufacturing plant are expected to be very low as a result of these precautions.

Carbon fiber released during prepregging is almost entirely associated with processing steps prior to resin impregnation. These include the pouring of chopped fibers into the extruder blender and kreel feeding to the islet board prior to solution coating. Both processes, which involve only a small percentage of total production, are controlled by work station hoods. The only other emission points are associated with fiber pullout during cutting or chopping. Exhaust hoods are also employed in these areas.

Finally, carbon fibers are released from composites as a result of final shaping, including sawing, drilling and grinding. Control methods include water sprays and exhaust hoods with bag filters.

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The waste dust is sealed in polyethylene bags and is either disposed of in landfills or used as a filler for certain processes. There is no specific information concerning fugitive emissions; however, plant operators believe that carbon fiber emissions to ambient air are very low.

LABORATORY EVALUATION OF CARBON FIBER RELEASE

To develop some practical information regarding emissions of carbon fibers during finishing operations (drilling, sawing, grinding), samples of carbon fiber reinforced sportswear were obtained for laboratory evaluation. These are as follows:

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Tennis Racket - the frame and handle consist of an epoxy core wrapped with carbon fiber tape and an overlay of glass fiber tape (Figure 4).

Gold Club Shaft - consists of several layers of wrapped carbon fiber tape (Figure 5).

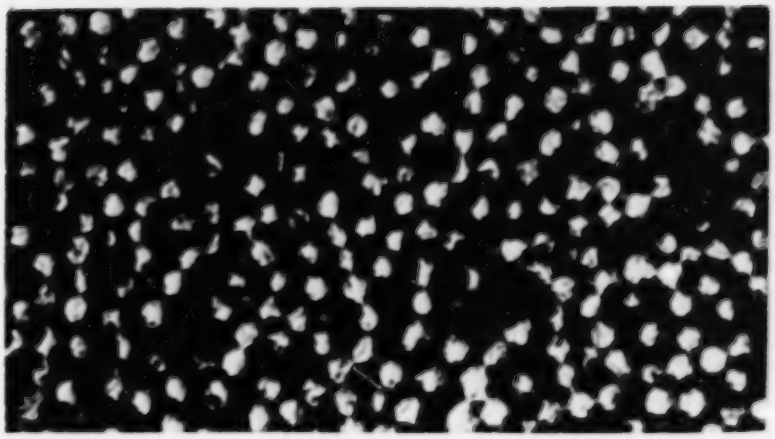
Machining operations (drilling, sawing, and grinding) were carried out in a glove box, with exhaust air being pulled through a two-stage filter consisting of 1 mm mesh size bridal veil backed by a 0.1 μm pore size Nuclepore filter. Each operation was performed for a period of two minutes. An exhaust blower operating at 50 lpm was run for five minutes prior to the start of the experiment and ten minutes after the end to withdraw all airborne particulate matter. At the end of a run, the bridal veil was put between two sheets of mylar (at which point many of the collected particles transferred to the mylar by electrostatic attraction) and the Nuclepore filter put in a cassette for subsequent optical viewing. The machining wastes were collected and weighed to provide a rough estimate of particulate emissions, assuming that the airborne dust content is proportional to weight loss. The relative weight loss ratios compared to that for golf shaft drilling were as follows:

	Relative Weight Loss From Machining	
	Golf Shaft	Tennis Racket Handle
Drilling	1	7
Sawing	4	4
Grinding	12	6

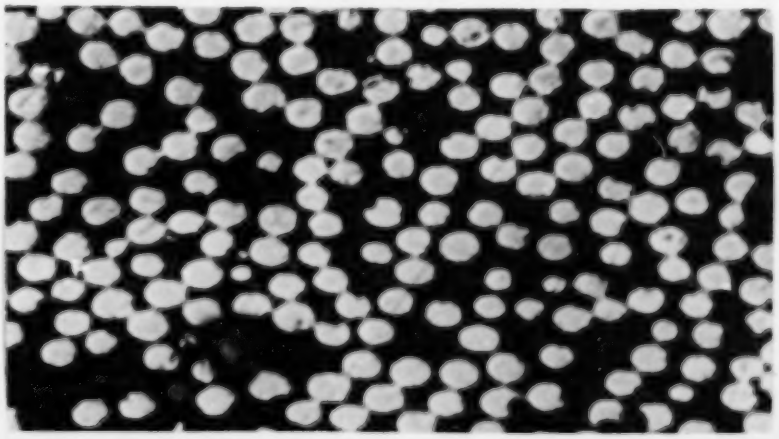
Photographs of the mylar-coated bridal veil associated with golf shaft grinding are given in Figure 6. It is noted that most of the carbon fibers are in the range of 0.1 - 0.3 mm in length, with the longest fiber observed being 1.8 mm in length. The backup Nuclepore filters, shown in Figure 7 for the three types of machining operations carried out on golf shafts, reveal only a few fibers, with most of the particles being isotropic, presumably particles of resin. The relative densities of particle loading are noted to increase markedly for sawing and grinding as compared to drilling.

Grinding experiments were repeated on both the golf shaft and tennis racket handle, with airborne particulate collected on a Nuclepore filter. Grinding times were reduced by 5 and 10 times, respectively, for these samples to prevent filter overloading. These filters were examined by scanning electron microscopy. Essentially no fibers were observed for the tennis racket handle sample, and particulate levels were very low. For the golf shaft, numerous particles having sizes of 5 - 25 μm in extent were observed, with ~10 percent being fibers. Most fibers were short (less than 50 μm), with aspect ratios of 5 or less. Typical particles are shown in Figure 8a, with a single fiber shown at higher magnification in Figure 8b. There was no evidence of fiber splitting. It is concluded that fibers emitted from machining operations are predominantly less than 0.1 mm in length. These results are in good agreement with those of Wagman et al (4).

(a)

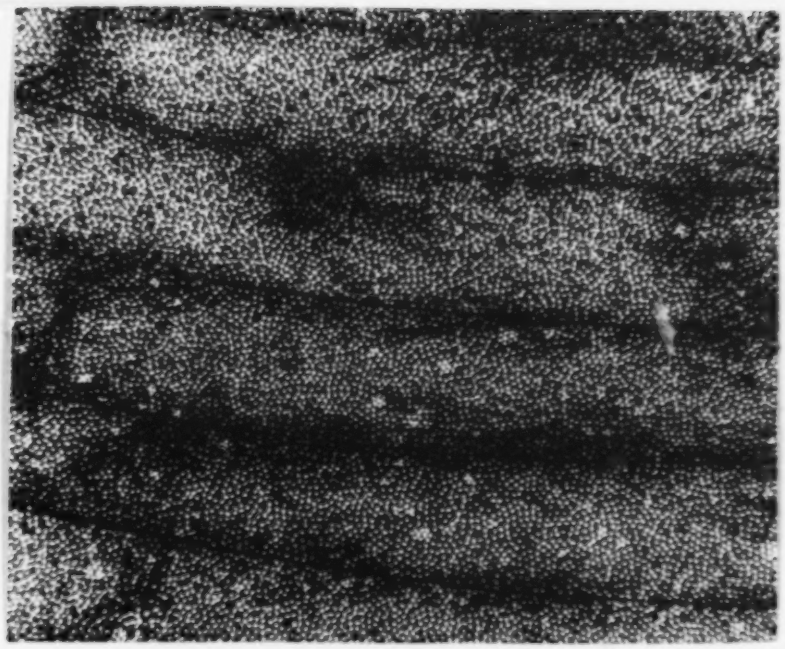


(b)

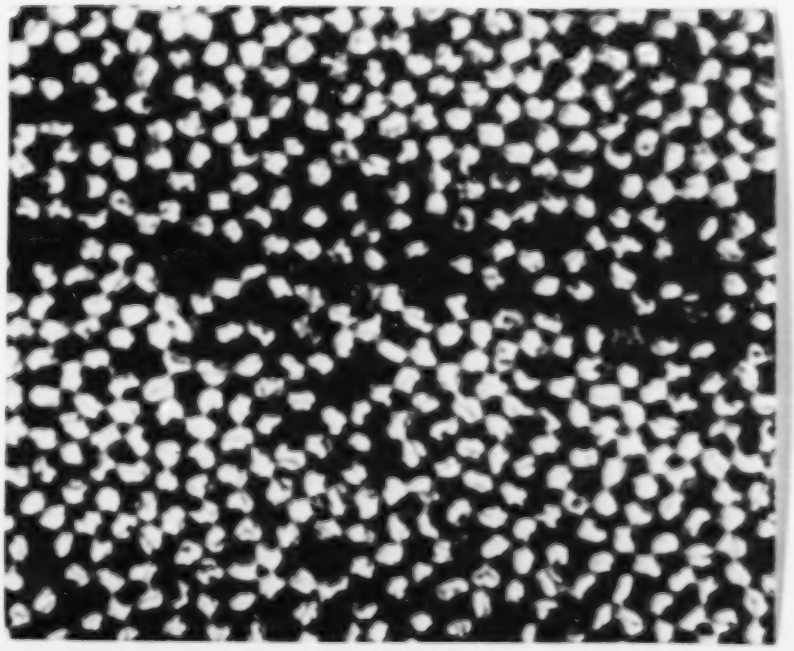


(c)

Figure 4. Cross Section of Tennis Racket Handle
a) Carbon Fiber Tape Wrapped with Glass Fiber Tape, 125X
b) Carbon Fiber Tape, 600X
c) Glass Fiber Tape, 600X

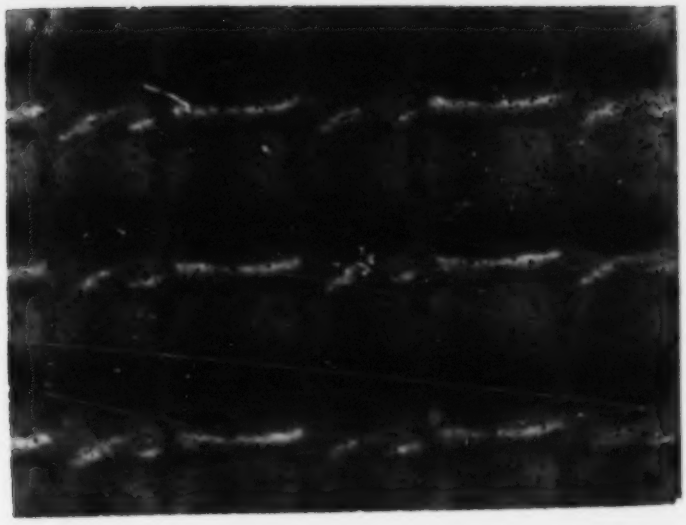


(a)

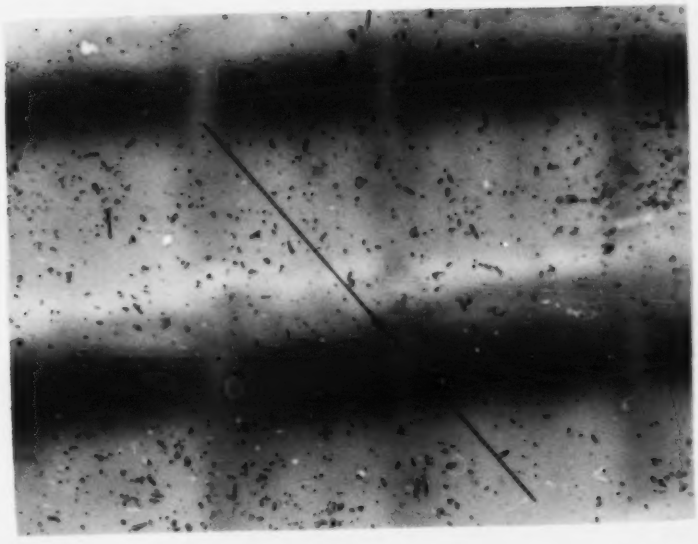


(b)

Figure 5. Cross Section of Golf Club Shaft: Carbon Fiber Tape Construction
a) 125X, b) 600X



20x



40x

Figure 6. Particulate Catch on 1 mm Mesh Bridal Veil for Golf Shaft Grinding

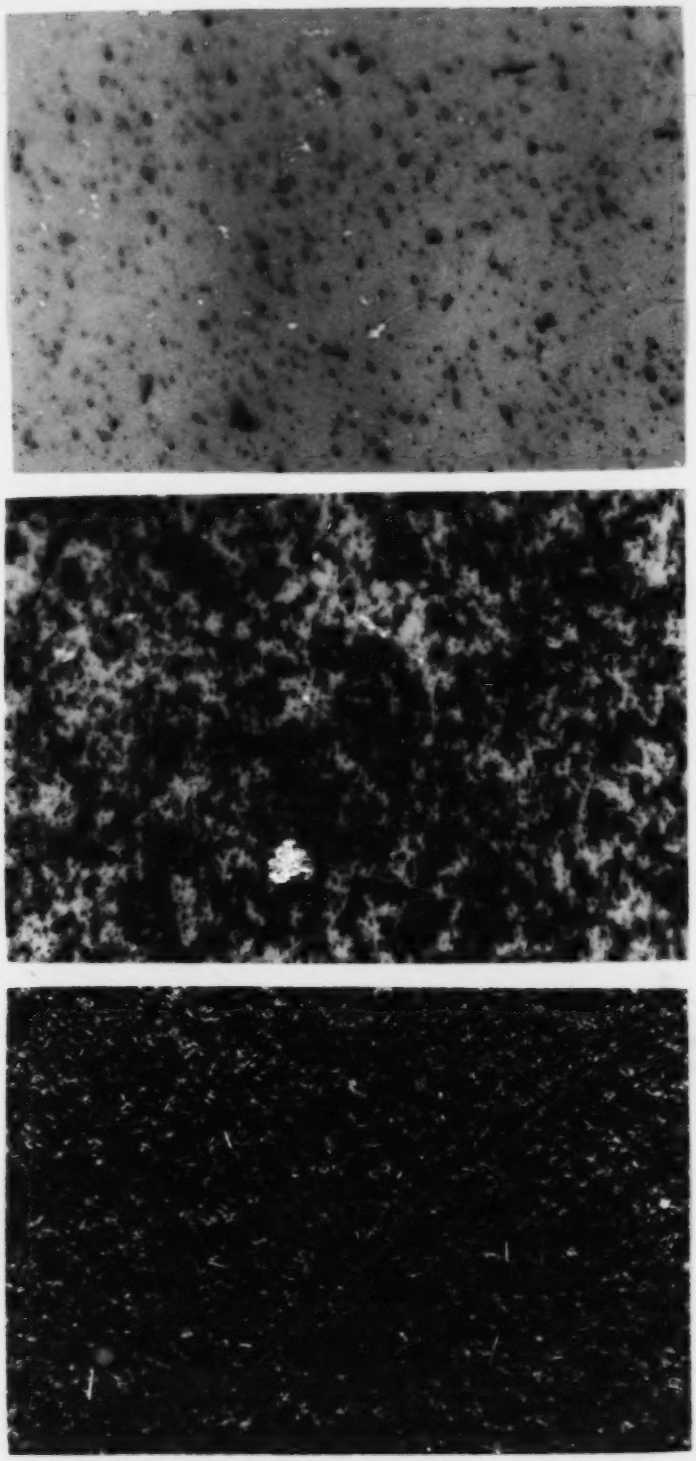
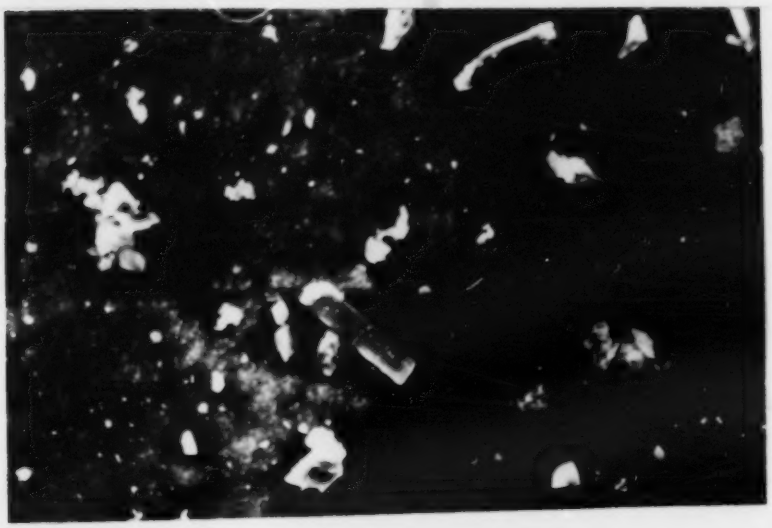
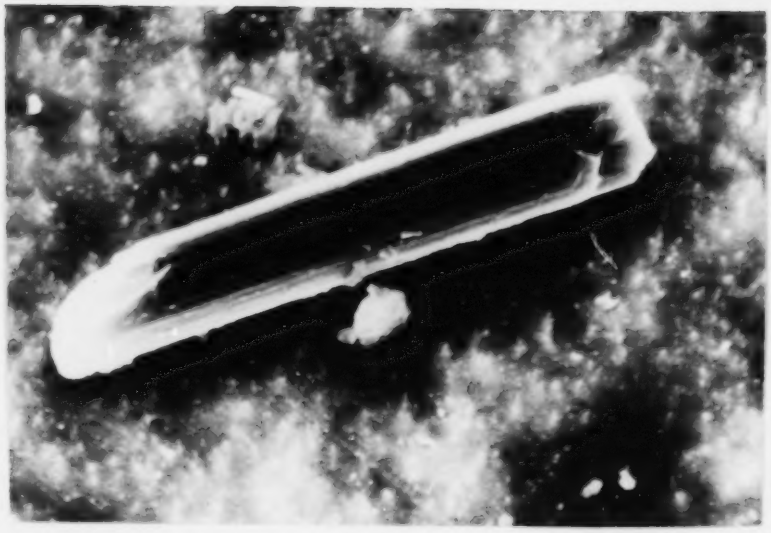


Figure 7. Particulate Catch on Nuclepore Backup Filters, 40x, for Machining Operations on Golf Shaft



a. 625x



b. 2500x

Figure 8. Scanning Electron Microscope Photographs of Particles Collected on a Nuclepore Filter During Golf Shaft Grinding: a) General View, b) Individual Carbon Fiber

CARBON FIBER EMISSION SCENARIOS

To permit a comparative ranking of monitoring devices and concepts on the basis of their ability to detect and quantify carbon fibers, it is necessary to estimate the characteristics of typical carbon fiber containing emission streams. On the basis of the carbon fiber manufacturing process review undertaken in this contract and previous studies of the emissions from municipal incinerators, three typical carbon fiber emission scenarios have been developed. They are summarized in Table 1.

Scenario A represents a carbon fiber textile weaving process conducted in an isolated room equipped with a HEPA filter air cleaning system. A typical system operates at 100 m^3/min and exhausts through a duct of about 8 m^2. The linear velocity is thus about 0.2 m/sec. The weaving room operates near ambient temperature, pressure and humidity and is subject to low total particulate loading due to filtered air recirculation. Manufacturing plant operators estimate that carbon fibers of about 0.5-2.5 mm in length are present in the room air at concentrations up to 10^7 to 10^8 fibers/m^3 as a result of partial fracturing of carbon fiber tows at points of high mechanical strain. The monitoring point could be anywhere between the emission point and the HEPA filter present.

Scenario B consists of a carbon fiber composite machining process, such as grinding, which may be required during formation of a commercial product, e.g., tennis racquet. Generally, emission of carbon fiber and other particulates from the machining station is controlled by localized air flow in a hood. The particulates collected by the hood system are frequently removed from the gas stream in a baghouse and ultimately disposed of in a landfill. An appropriately-designed ventilation system has a gas flow of about 5-20 m^3/min through a duct with a cross section of about 0.01-0.1 m^2. The linear velocity in such a case is quite low (e.g., 1-2 m/sec). As in the previous scenario, exhaust air is at ambient conditions. Much higher carbon fiber concentrations may be present here, and other particulates may also be present as well at a level about equal to the number of carbon fibers.

Finally, Scenario C involves the ultimate disposal of finished products, e.g., tennis racquets, in a municipal waste incinerator. Such a system exhausts a large volume of air (200-300 m^3/sec) with a very large concentration of particulate. However, it is obvious that only a small fraction of the waste material combusted contains carbon fiber composite. Therefore, in a cubic meter of effluent, only about 10^3-10^5 carbon fibers may be emitted in the presence of about 10^9 or greater particles. The carbon fiber size range expected in a combustion source is from 0.1-10 mm.

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TABLE 1. CARBON FIBER EMISSION SCENARIOS*

Parameter	Scenario A	Scenario B	Scenario C
Process	Textile Weaving	CF Composite Machining	Waste Disposal by Incineration
Control Procedure	Room Filtration	Hood/ Baghouse	?
Air Movement			
Gas Volume (m^3/min)	~100	5-20	200-300
Duct cross section (m^2)	~8	0.01-0.1	10-15
Linear velocity (m/sec)	0.2	1-2	5-10
Temperature (°C)	ambient	ambient	100-150
Moisture (%)	---	---	10-20
Total Particulate Mass (mg/m^3)	1	5-10	200-500
Corrosive Gases	no	no	yes
Maximum CF Concentration (number/m^3)	10^7-10^8	10^8-10^{10}	10^3-10^5
Average Length (mm)	5	0.5	1
Length Range (mm)	0.5-25	0.1-2	0.1-10
Presence of			
other particulate	no	yes	yes
other fibers	no	maybe	probably
other conducting fibers	no	no	maybe

Source: Arthur D. Little, Inc. estimates

*[Added Note: A reviewer of this final report brought to our attention the following measured data obtained on EPA Contract No. 68-02-3229.

	A	B
Total Particulate Mass (mg/m^3)	0.1-10	---
CF Concentration Range (number/m^3)	10^3-$5x10^4$	10^4-10^8
Average Length (mm)	1.5	0.1

The implication of these data to candidate carbon fiber monitor methods is indicated by broken circles in Figure 28.]

SECTION 6

EVALUATION OF MONITORING METHODS

CANDIDATE METHODS

Most of the instruments potentially useful for continuous measurement of carbon fiber mass and number were developed in support of classified government programs to determine fiber release rates in association with fire and explosion in simulation of an aircraft accident (5,6,7). In addition, there are several methods developed for other types of fibers, such as asbestos, which may be adapted to the measurement of carbon fibers. In general, the test environment of concern in this study, e.g., manufacturing facility, differs from that of explosive combustion sources in terms of emission duration, fiber concentration and length of fibers. It is possible, however, to extrapolate performance data from previous studies to requirements for a continuous monitor for carbon fibers in the manufacturing environment.

Various instrumental methods that have been utilized for carbon fiber measurement or that have potential as a measurement method are listed in Table 2. These may be grouped into categories of contacting, locally sensing and remote sensing.

Contacting Detectors

These detectors interact with fibers in their immediate vicinity. The devices depend on the conductive nature of carbon fibers and the length of the fibers, i.e., generally >0.1 cm. They provide detection in terms of the number of fibers within limited sensitive spatial volumes. As a result, information on deposition rate and linear velocity of the air stream must be obtained to calculate fiber concentration.

Ball Gauge--

One of the physically (but not necessarily electronically) simplest devices is a conducting ball (Figure 9). As discussed theoretically by Bucher (8, 9), a conducting ball mounted on an insulated column and connected to a high voltage direct current source (2000 V) generates a diverging electrostatic field in its vicinity. Close to the charged metal ball (i.e., within several times its diameter), the electric field induces a dipole moment in conductive carbon fibers. Carbon fibers coated with non-conducting resins and some other fibers also develop a dipole moment. Most non-conducting fibers do not respond but adsorbed water or surface contaminants may facilitate production of a dipole.

TABLE 2. CANDIDATE MONITOR METHODS

- Contacting
  - Ball Gauge
  - Electric Grid - Arc
  - Electric Grid - Resistance
- Locally Sensing
  - Optical
    - Opacity
    - Scattering
      - Fiber Aerosol Monitor (FAM)
      - Near Forward Scattering
      - Rotating Lens
  - Microwave
    - Interception
    - OSGEF
- Remote Sensing
  - Radar
  - Infrared

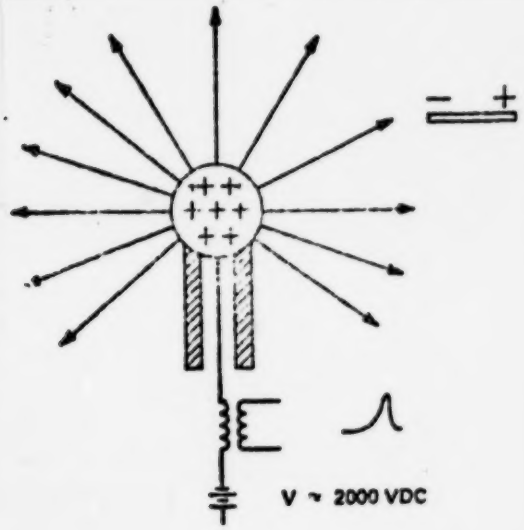


Figure 9. The Ball Gauge Electronic Discharge Detector (7)

Spatial gradients in the electric field produce translational forces on the conductive fibers which are then attracted to the surface of the ball. Upon contact, they acquire a like charge and are then repelled. This process generates a current pulse, the intensity of which can be recorded with suitable electronic circuitry. The number of pulses is proportional to the number of fibers and the amplitude of the pulse has been shown to be proportional to the fiber length (10). The device is sensitive to carbon fibers greater than 3 mm in length, with smaller fibers generally lost in the background electrical noise. The method may be modified to detect slightly smaller fibers.

Other limitations of the method include the fact that it cannot distinguish between carbon fibers and other conductive fibers, and it is sensitive to leakage currents resulting from dirt accumulation on the ball support insulators. Of more concern is the fact that the sampling volume is sensitive to fiber length, voltage and air velocity. Specifically, the sampling volume increases for longer fibers and decreases with higher velocity. Thus, the data must undergo pulse height analysis correction to provide accurate measurement of size distribution of mixed length fibers.

When placed in a flowing stream of carbon fibers, the high voltage ball can detect an exposure level of about 10^4-10^7 fiber-seconds/m^2 which, for duct flow, corresponds to 1-100 fibers/ml. In the presence of very large numbers of fibers, the device can saturate. It is also apparent that a clump may appear as a single fiber.

Electric Grid--

Electric grids, which are similar in design to common "bug zappers", consist of a grid of parallel conductors of alternate polarity. The spacings between conductors is made as small as practical so that fibers falling on the grid will connect two conductors.

Arc-Type - In the high voltage type (100-1500 volts) grid (11) (Figure 10a), a conductive fiber is vaporized and a current pulse generated. Fibers smaller than the grid openings may not impact the grid and thus will not be detected. Of course, an oblique orientation of fibers with high aspects ratios is critical to detection since the diameters of most carbon fibers are less than 10 μm. The grid array can be made quite large to intercept a large area. It is apparent that some foreign matter may accumulate on the device. False pulses may occur due to matter such as insects and also as a result of moisture condensation. If the grid is operated above the dew point, the latter will not be a problem. The method is not length specific except for a lower detection limit and suffers from decreased accuracy as material accumulates on the mesh. Also, it cannot distinguish carbon fibers from other conductive fibers.

Resistance-Type - Grids operated at a lower voltage (<20V) do not vaporize carbon fibers which come in contact but generate a change in resistance of the array (7) (Figure 10b). The resistance change and an increase in current as fibers accumulate is a function of fiber type and length, but little experience has been gained in understanding the complex relationships in fiber concentration, size and resistance. Since the carbon fibers accumulate on the grid, its character changes and it becomes inoperative at some point without cleaning. If fibers are blown off the grid, a change in resistance occurs.

Locally Sensing Devices

These detectors are similar to contacting detectors in that fibers pass through a sensing volume immediately adjacent to an energy source and detector. However, fibers only pass through the beam of electromagnetic energy and absorb or scatter some of the energy, thus reducing the energy received by a detector. In the case of a device which absorbs energy, the reduction in energy as received by the detector is proportional to the concentration of particles as in Beer's Law. The obscuration is normally reported in stationary source plumes as percent opacity.

Optical Methods--

Opacity - A light emitting diode source/detector (LED) device has been used to measure fibers in a plume (12). The device consists of a collimated LED light source, focusing lens, neutral density and band pass filters and a silicon pin diode detector (Figure 11). The device measures the number rate of all particles passing through the light beam. The number concentration is calculated on the basis of air velocity and beam volume. The mass concentration can be determined only if particle size distribution is known. If a fiber passes through the beam, the

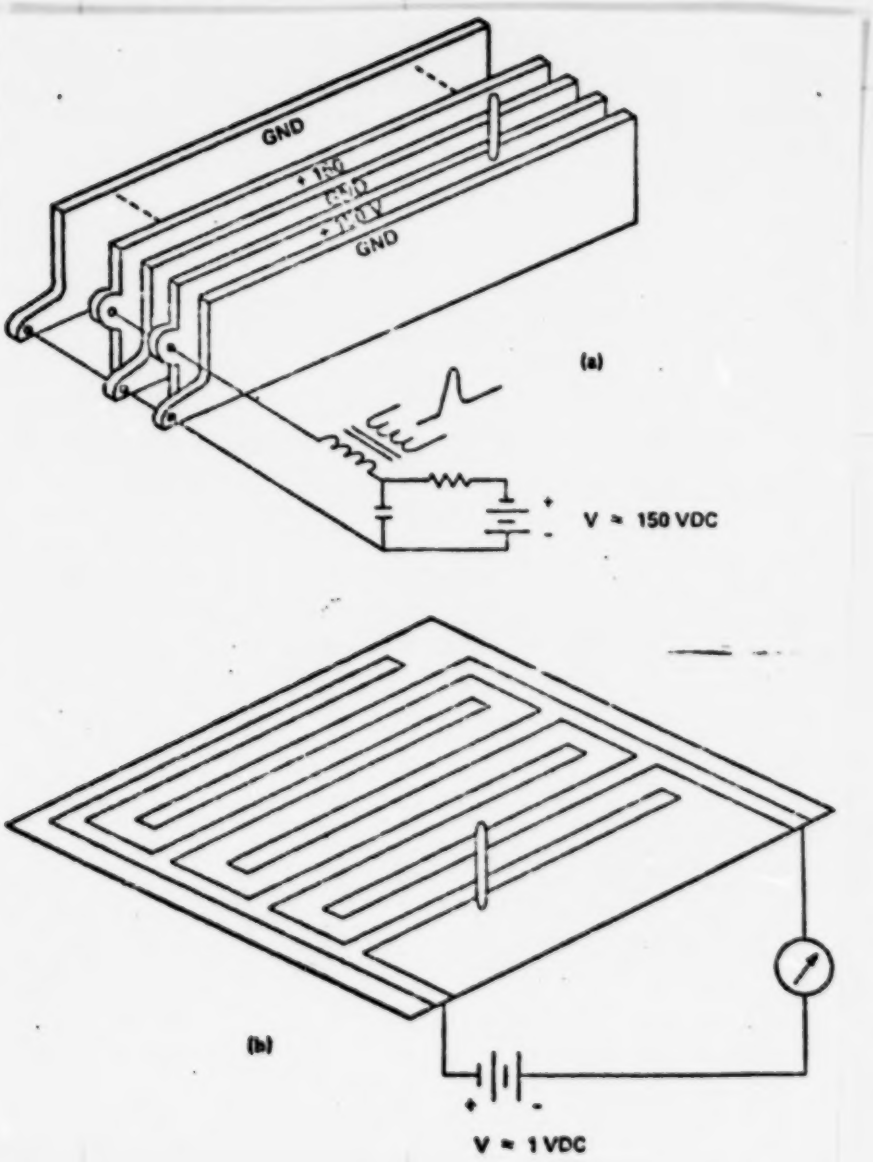


Figure 10. Locally Contacting Electric Grids:(7)
(a) Arc Detector and (b) Resistance Detector

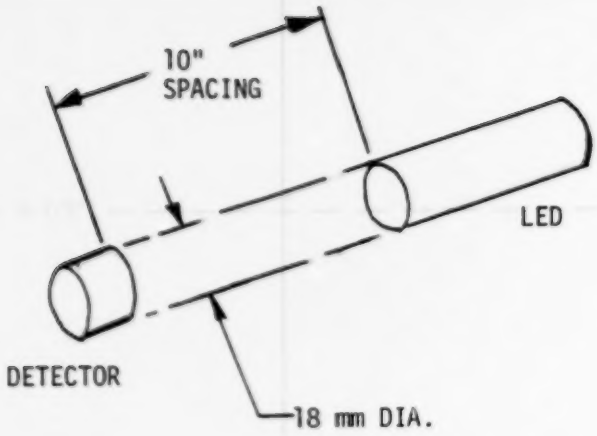


Figure 11. LED Particle Detecting System(7)

light obscuration is detected as a pulse of measurable width. The number of pulses is a measure of the number of fibers which traversed the beam. Measurements become saturated when the pulses are too numerous to resolve. Disadvantages of this method are that all types of particles are detected and a small beam volume may result in a small detection area.

Scattering - Optical devices, which utilize light scattering as the active measurement principle offer considerable advantage over opacity devices since they differentiate between particles and fibers. A fibrous aerosol monitor (FAM) was recently developed (13, 14) to provide real time measurements of fibrous particle concentration. Other conceptually feasible solutions have been and continue to be explored (15, 16).

The FAM (Figure 12) was developed to be a practical, field-worthy instrument for detection, counting and sizing of acicular-shaped particles even in the presence of nonfibrous particle concentrations several orders of magnitude higher than those of the fibers of interest. The monitor senses fibers by (a) inducing fibers to rotate rapidly by application of a rotating high-intensity electric field and (b) detecting the light scattering signature associated with these fibers as they are illuminated by a monochromatic and parallel light beam. As fibers flow through the sensing region, they are illuminated by a laser beam parallel to the laminar flow. Scattered pulses resulting from induced fiber oscillation are detected perpendicular to the beam axis. Thus, two light scattering peaks are detected for each fiber rotation. The signal pulse sharpness is a function of fiber length and longer fibers produce narrower pulses than shorter ones.

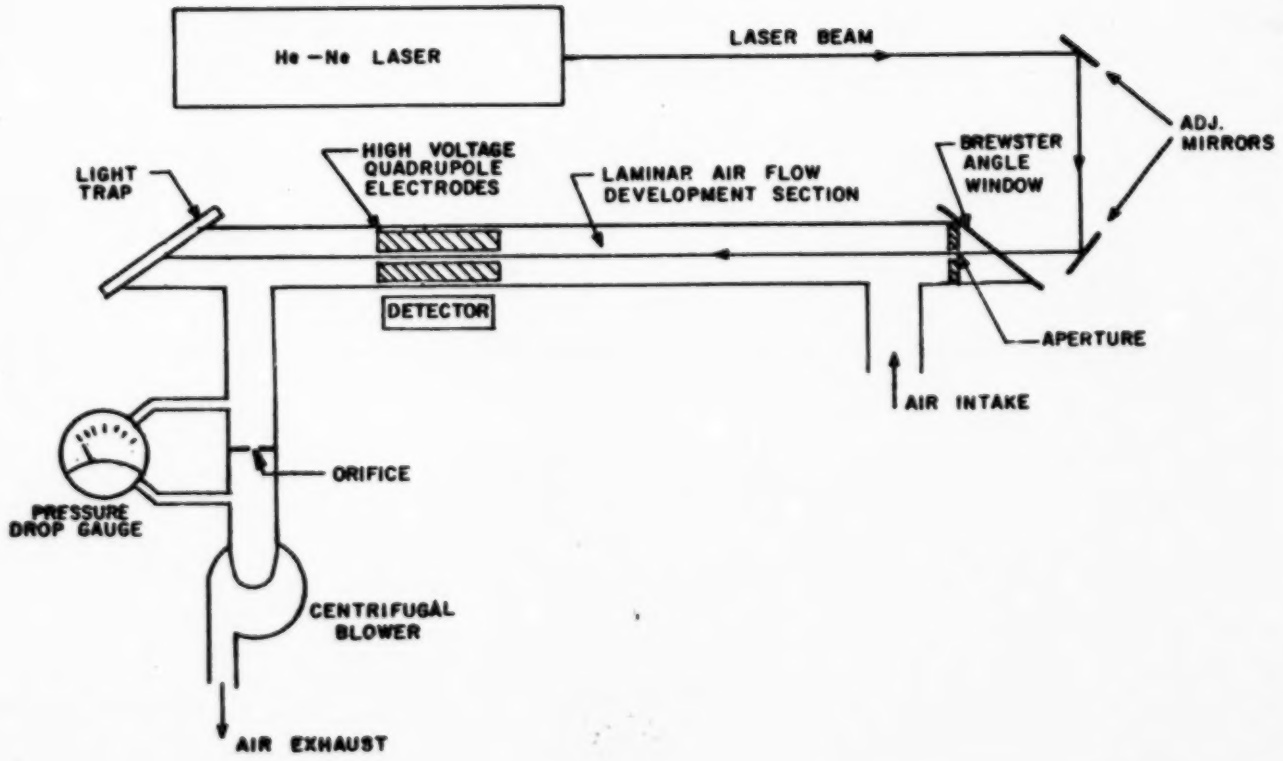


Figure 12. Schematic of optical and flow system of FAM (13)

The device's fiber criteria are met if: (a) the signal pulses are synchronous in frequency and phase with a reference waveform, and (b) the average pulse amplitude, the ratio of pulse amplitude to signal average and the duration of the pulse train meet certain preset values. The device can detect fibers greater than about 2 μm in length and 0.2 μm in diameter in the presence of particle concentrations exceeding those of fibers by up to 10^6. At present, the portable device can count fibers greater than a certain length and with an aspect ratio >3:1 but does not supply size distribution data. Also, the size of the sensing chamber limits the upper detection limit fiber length to about 1 mm due to the limited size of the light beam. Nonconductive fibers contain enough adsorbed water or impurities to align in the electric field and thus are counted.

A two-detector, aerosol-fiber counter exploiting near forward scattering has been operated in a laboratory environment (15) (Figure 13). The device measures the intensity of a diffraction pattern of a fiber at two locations. The intensities are different for a fiber but not a near spherical particle. By selection of a threshold ratio of intensities, a length to diameter criteria for a fiber can be set. Fibers of known orientation are most easily detected, but even randomly-oriented fibers can be detected with a certain probability. The efficiency of fiber counting can be enhanced by aligning fibers to maximize the ratio of light intensities. Two effects, i.e., use of laminar flow in a narrow tube and use of an electric field up to 3000 V/cm in the same direction as flow reinforce each other. The flow restriction alone produces about

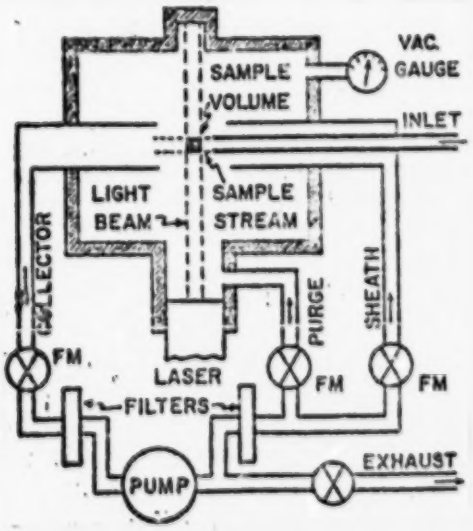


Figure 13. Schematic of two-detector, aerosol fiber counter (15).

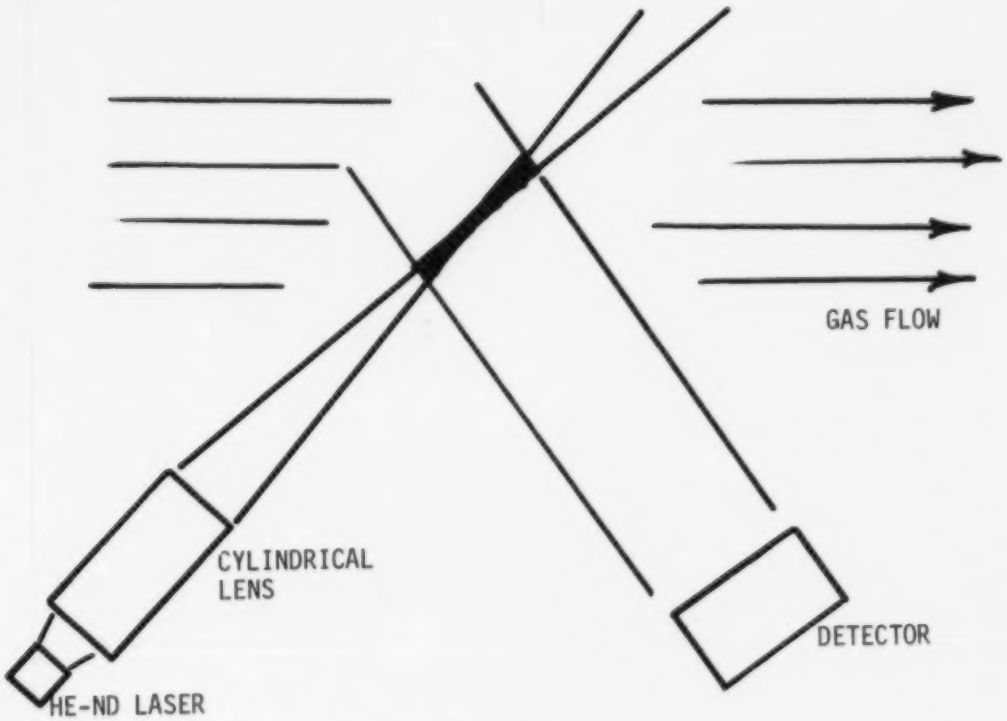
20% alignment for asbestos fibers while the flow and electric field produces about 35% alignment. The background light in the scattering chamber sets the lower size limit that can be classified and the saturation level of the photometers determines the upper size limit. The instrument discriminates against common particles, such as room dust, chalk dust and cigarette smoke, but glass wool fibers are not detected perhaps due to fiber length. The instrument is still under development, and performance characteristics are not very well defined. Tests with asbestos indicate a detection range of 10^6-10^8 fibers/m^3 in a dust background of up to $5x10^9$ particles/m^3.

A fiber monitoring concept has been disclosed (16) in which light from a He-Nd laser is passed through a rotating cylindrical lens and focused to a slit. The slit size is selected to have a ℓ/ω of 5 or greater with the width somewhat less than the length of fibers to be measured. An aiming detector at an appropriate scattering angle to the direct beam is set to look at a specific "window", i.e., the volume of space corresponding to the waist region of the slit. This geometry is shown in Figure 14a. In practice, the focusing lens must rotate at an angular velocity somewhat faster than the linear velocity of the stream being monitored so that there are one or more rotations of the slit as a gas-stream particle passes through the window. In this way, non-fibrous particles have a constant scattering intensity whereas fiberous particles have variable scattering intensity due to losses from the fiber ends when the slit is rotated perpendicular to the fiber. This effect is shown schematically in Figure 14. The detected signal can be modulated to discriminate non-fiberous particles, with the remaining pulses corresponding to fibers analyzed to provide counts and perhaps dimensions (or aspect ratio) and mass. The method has the advantage of being able to "look" into a flowing stream. However, it cannot distinguish carbon fibers from others. It must be recognized that the method is a concept and requires further evaluation.

Microwave Methods--

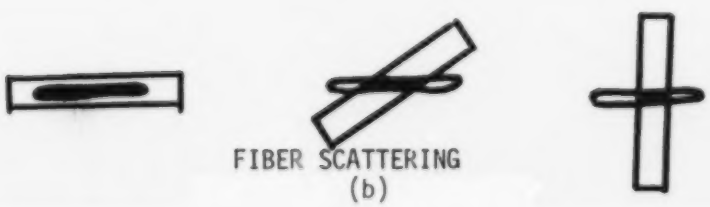
Interception--Electromagnetic radiation in the microwave region can be used to make opacity and scattering measurements analogous to optical methods (7). A microwave system has been developed to measure the mass flux of clusters and clumps of fibers as well as single fibers. It consists of 12 Gigahertz source, transmitter horn and collimator lens in one column, and a lens, receiver horn and detector/amplifier in the other column (Figure 15). As fibers pass through the beam, the energy received is reduced in proportion to the fiber mass. The beam length and diameter are about 62 cm and 9 cm, respectively. The size is determined by optimization of path length with wave length of the beam. The microwave detector responds to nonconductive materials as well as fibers and thus is not particularly selective. It also requires high concentrations of large clumps to operate; in fact, it operates at levels where other devices, electrical or optical, saturate.

In a more sophisticated system, the microwave frequency is varied through a broad range so that individual fibers of varying length are electrically resonated. Such scanning improves the sensitivity of measurement by providing



(a)

PARTICLE SCATTERING



FIBER SCATTERING

(b)

Figure 14. Fiber Monitor Concept Based on Rotating Slit Optical Scattering (16).

data at the absorption and/or scattering cross section maxima of the fiber. However, the scanning method is slow, and only a small fraction of the fibers within the sample volume can be counted at any one time. Furthermore, the equipment is expensive and bulky if the frequency scan is to be made wide enough to record any reasonable portion of the naturally occurring fiber length distribution. As with optical methods, these microwave approaches cannot distinguish between conductive fibers of different materials.

OSGEF--Another method recently proposed for carbon fiber measurement is based on the establishment of a unique optical signature resulting from the interaction of a conducting fiber with a high frequency electric field (17). The generalized approach is illustrated in Figure 16. The principle of operation depends on the perturbation of equipotentials by a conducting fiber in an electric field (Figure 17) and the ability of the conductor to adjust its surface charge distribution rapidly enough to maintain the surface equipotential. There are two important features about the electric field distribution near a fiber. First, there is an extremely large increase in the electric field strength at the ends of the fiber. Second, there is a substantial electric field perpendicular to the fiber along its entire length except at its middle. These observations are important in that they lead to the ability to produce optical signatures by two methods which are highly selective in that the signature depends on both the length to diameter ratio and the conductivity of the material measured. The first method would use the electric

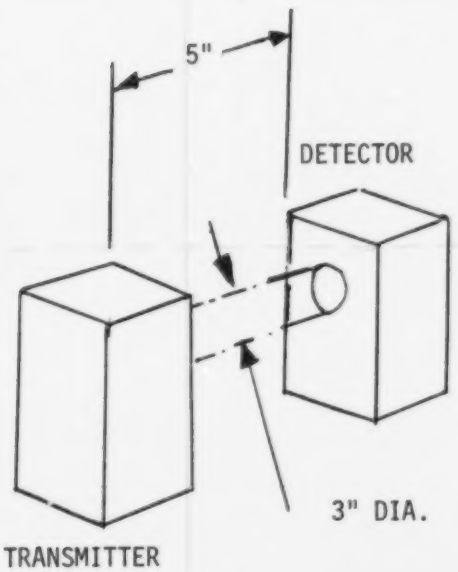


Figure 15. Microwave System (7)

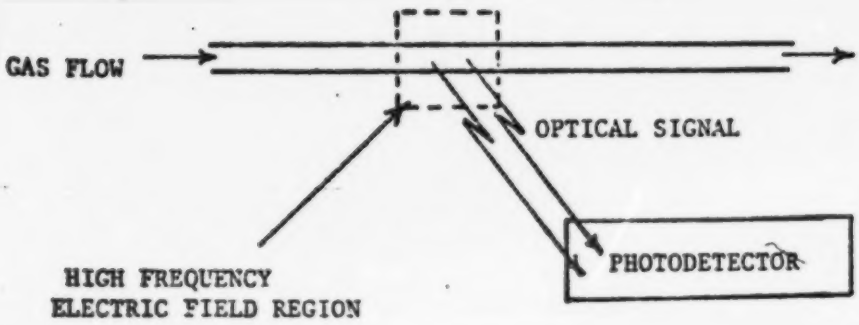


Figure 16. Elements of Carbon Fiber Measurement Apparatus Based Upon Optical Signature Generation in a High Frequency Electric Field

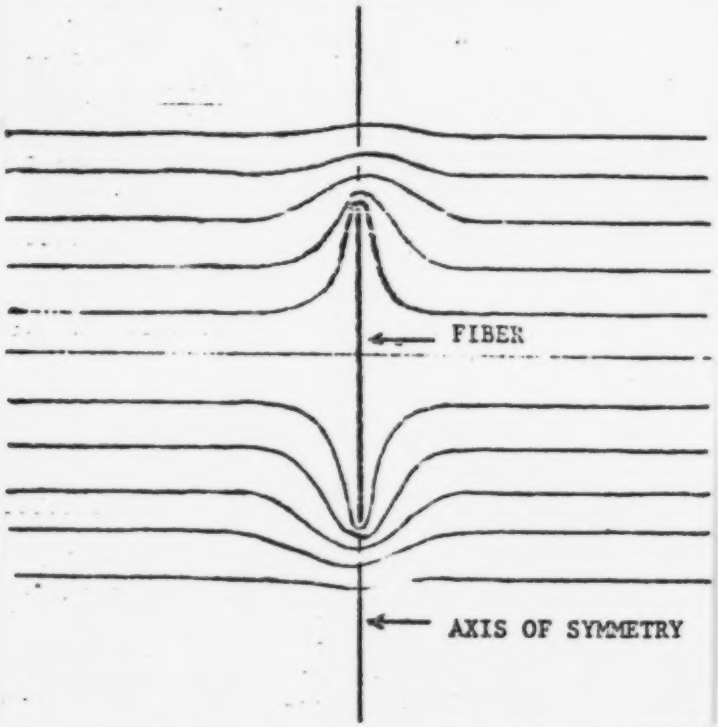


Figure 17. Equipotentials Around Conducting Fiber Parallel to Electric Field

field enhancement at the ends of the fiber to raise the local field above the ionization threshold of the surrounding sample gas, thereby creating a glow discharge at the fiber ends which may be optically detected. The second method would be based on an optical measurement of the enhanced thermal radiation from fibers. The elevation in fiber temperature which produces this enhanced thermal radiation is a direct result of the electric field perpendicular to the length of the fiber.

The present study was briefly extended to permit a preliminary laboratory evaluation of the OSGEF concept. These studies are described in a subsequent section of this report.

Remote Sensing Devices

The last category of carbon fiber monitors are based on devices which detect fibers at a distance. Two concepts include active radar and passive infrared instruments (Figure 18). Their prime utility is to identify the physical extent of a dense plume of particles. In the case of the radar

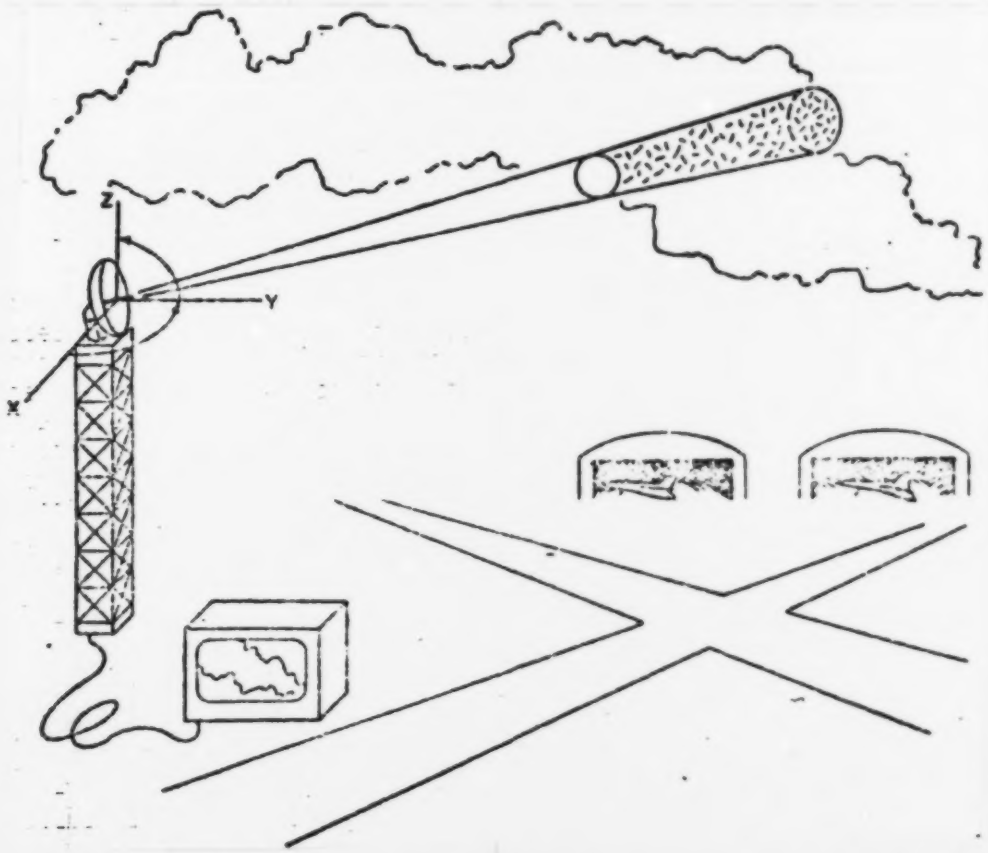


Figure 18. Remote Sensing Devices: Active Radar and Passive Infra-Red. (7)

concept, a narrow beam of microwave energy can reflect from a cloud of adequate radar cross section. Since carbon fibers and other matter have radar cross sections of unknown magnitude, detection of other than very dense plumes of only carbon fibers is not feasible. The infrared detection concept consists of passive detection of the thermal radiation from a cloud of carbon fibers. The optics consists of a small scanning light collecting aperture with an angular field similar to that used for radar detection. It is quite apparent that changes in thermal background radiation occurring during angular scanning present a serious threshold problem. Of course, clouds of all particles, not just carbon fibers, yield a detectable signal.

LABORATORY EVALUATION OF OSGEF CONCEPT

Introduction and Summary

A brief experimental investigation was undertaken to quantify the behavior of carbon fiber in a high frequency electric field and establish the level of optical/thermal signal generation which can be achieved and applied to carbon fiber detection.

This method has been given the acronym OSGEF, for Optical Signal Generation in a high frequency Electric Field, and an instrument based upon this approach would have the elements previously shown in Figure 16. Here a sample of the gaseous effluent under study is directed through a high frequency electric field region where any carbon fibers present are selectively heated. The accompanying increase in infrared emissions from the fiber is received by the photodetector, producing a signal which can be related to the number density and mass flow of the carbon fiber.

In general, the validity of the OSGEF concept has been verified. The method is quite specific to carbon fiber, since fibers with significantly greater or lesser conductivity do not produce comparable optical/thermal signals. The method is also quite sensitive, allowing single fiber detection of lengths as short as 1mm with good signal to noise ratio. Lengths of tow, or clumps of fibers, produce larger signals in proportion to the exposed area.

A possible limitation to the OSGEF method is imposed by the time required to heat the fiber and generate the desired optical/thermal signal. This heating time depends upon the heat capacity of the fiber and the rate of heat loss through conduction, convection, and radiation. For the 10 μm diameter carbon fiber used in our measurements, the time constant for fiber heating was approximately 100 μsec., yielding an optical/thermal emission rise time of about 20 msec. Thus the carbon fiber must be exposed to the high frequency field for an appreciable fraction of 20 msec. in order to produce a nearly full amplitude signature. However, less than full amplitude signals are still useful because of the sensitivity of the method, as demonstrated by the good results obtained with 6 msec. excitation periods in the present work.

Based upon the ruse of a 1 cm r.f. excitation gap operating at 2450 MHz and an indium arsenide optical/thermal detector, 1 mm and longer carbon fibers can be detected with a signal to noise ratio greater than 10:1 for r.f. field strengths of 135 volts/cm or greater, providing the

fiber velocity through the sensing region is kept under 100 cm/sec. Individual fibers or fiber clumps may be counted up to a rate of about 100/sec, and the average mass flow will be proportional to the average signal level as long as the fiber density is not so high as to cause significant shadowing within the sensing region. Typical power dissipation within a fiber which extends through a 1 cm r.f. gap with a field strength of about 170 volts/cm is on the order of 1.5 watts.

Methodology and Measurement Results

Two separate experiments were designed for the present study. The first was intended to provide a measurement of the thermal time constant of the carbon fiber and to calibrate the emitted optical/thermal signal in terms of the power input to the fiber. The second was designed to quantify the microwave/fiber interaction and to establish the design parameters for practical OSGEF operation.

Fiber Heating and Optical/Thermal Emission Experiment--

All of the studies were conducted using a representative pitch-precurser carbon fiber. This was Union Carbide, "Thornel" P-55S, grade VSB-32. A data sheet for this material is attached as Table 3.

Figure 19 diagrams the apparatus used for the study of fiber heating and optical/thermal signal generation. The fiber was directly heated by the pulse generator output through silver epoxy contacts to the fiber ends. Mounting the fiber was a delicate process and was accomplished by extending the fiber over the top of two upright brass posts rigidly mounted in a teflon block. Each of these posts had a shallow groove in the exposed end which received the fiber and maintained it in alignment with the adjacent post.

After positioning the fiber, silver epoxy was applied to the grooved post ends and allowed to air dry. The resulting contacts added negligible resistance to the fiber circuit and provided all the mechanical support required.

The mounted fiber was viewed by an indium arsenide detector placed 1 cm from the center of the fiber and inclined at an angle of 45^{o}. This

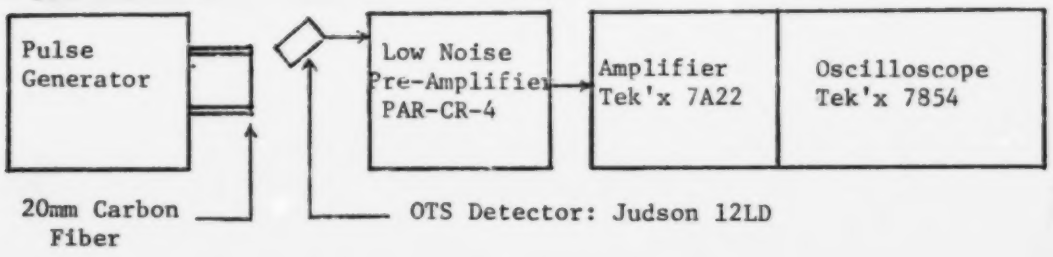


Figure 19. Signal Calibration Experiment

TABLE 3. THORNEL P55S CARBON FIBER DATA SHEET

TECHNICAL INFORMATION

UNION CARBIDE CORPORATION
CARBON PRODUCTS DIVISION
270 Park Avenue, New York, N.Y. 10017

BULLETIN NO. 465-245

"THORNEL" P-55S CARBON FIBER GRADE VSB 32-S

DESCRIPTION

"Thornel" P-55S Carbon Fiber Grade VSB-32-S is a continuous filament, high modulus strand from a pitch precursor. Grade VSB-32-S fiber is treated to provide a shear strength of 8000 psi in an epoxy composite and designed for use in stiffness-critical applications

TYPICAL PROPERTIES AND CHARACTERISTICS "THORNEL" FIBER GRADE VSB-32-S

PROPERTY	U.S. CUSTOMARY UNITS — VALUE		S.I. UNITS — VALUE	
Tensile Strength	$lb/in^2 \times 10^3$	275	GPa	1.90
Tensile Modulus	$lb/in^2 \times 10^6$	55	GPa	380
Density	lb/in^3	0.072	Mg/m^3	2.0
Filament Diameter	μ	10	μm	10
Elongation at Break	%	0.5	%	0.5
Elastic Recovery	%	100	%	100
Carbon Assay	%	99+	%	99+
Surface Area	m^2/g	1	m^2/g	1
Thermal Conductivity	BTU-ft/hr(ft^2)(°F)	58	W/m K	100
Electrical Resistivity	Ohm-cm $\times 10^{-4}$	7.5	μohm-m	7.5
Longitudinal CTE at 70°F (21°C)	PPM/°F	−0.5	PPM/K	−0.9
Specific Heat at 70°F (21°C)	BTU/lb°F	0.22	j/kg K	925

TYPICAL STRAND PROPERTIES "THORNEL" FIBER GRADE VSB-32-S

PROPERTY	U.S. CUSTOMARY UNITS — VALUE		S.I. UNITS — VALUE	
Yield	yd/lb	1600	m/g	3.23
Denier	g/9000m	2800	g/9000m	2800
Dry Pull Strength	lb	18	N	80
*Twist	tpf	0	tpm	0
Filaments/Strand	—	2000	—	2000
Fiber Area in Yarn Cross Section	$in^2 \times 10^{-5}$	24	mm^2	0.155

*Also available with a twist of 9 TPF (30 TPM)

THORNEL is a registered trademark of Union Carbide Corporation.

detector positioning was used to replicate that employed in the microwave/ fiber interaction studies. Figure 20 shows the sensitivity and typical electrical characteristics of the Judson 12LD detector selected for the present work. It is an inexpensive, room temperature device which was available in a convenient BNC connector mounting. This diode has an internal impedance of about 1000 ohms and was well matched by the low-Z input of the PAR model CR-4 low noise preamplifier.

TYPICAL ELECTRICAL CHARACTERISTICS

Operating Temperature:	300°K
Peak Response:	3.4 μm
Photon Efficiency:	>25%
Capacitance @ V_T:	15pf
Frequency Response @ V_T:	100MHz
Peak Responsitivity:	.6A/W
Test Voltage (V_T):	1V
Sensitive Area:	.02mm^2

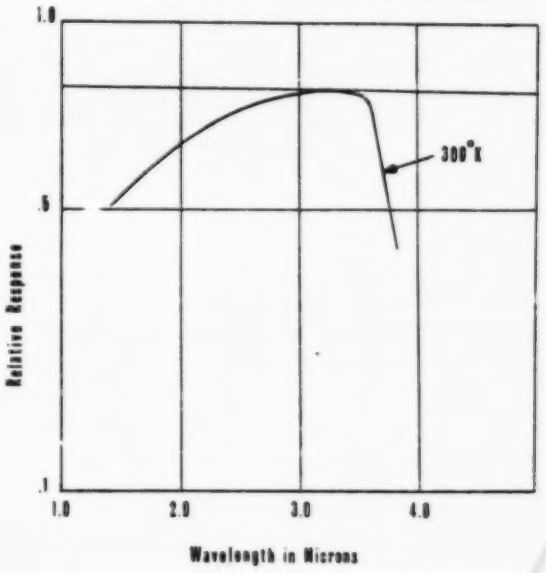
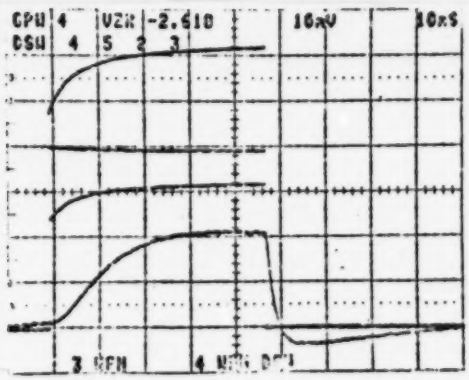


Figure 20. Characteristics of the Judson Laser Detector J-12LD

In view of the complex fiber viewing geometry, the wavelength dependent sensitivity of the detector, and the variation in the black body emission spectrum of the fiber as it is heated, no attempt was made to analytically relate the optical signal to the increase in fiber temperature.

All data collection was accomplished by means of a Tektronix Model 7854 waveform processing oscilloscope, using a 7A22 differential input amplifier and a 7B20 time base. Representative waveform traces for the pulsed fiber heating and optical/thermal signal generation experiment are given in Figure 21.

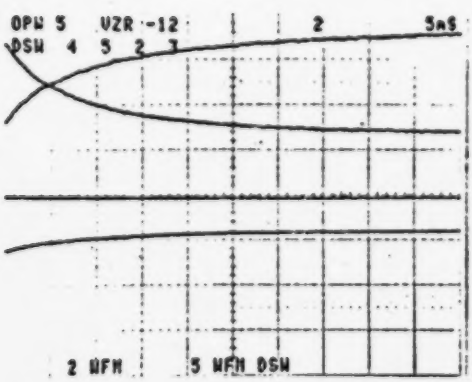


- Fiber Power Dissipation: 50mW/div
- Fiber Voltage Drop: 5V/div
- Fiber Current: 5mA/div
- Optical/Thermal Signal: 10mV/div

0 Reference: -3 div from display center

Figure 21. Carbon Fiber Pulse Heating and Optical/Thermal Signal

The optical thermal signal shown has a rise time of 19.0 msec. and a fall time of 2.0 msec. Figure 22 provides an expanded view of the heating pulse parameters and shows, in addition, the behavior of the fiber resistance during the pulse. The negative temperature coefficient of the resistance indicates that the fiber carbon content prevails over the graphite content in determining the resistance behavior. If the graphite dominated, a positive temperature coefficient would be seen. Resistance behavior can also be used to obtain an average fiber temperature rise during the pulse, provided a separate, oven calibration is made of the resistance vs. temperature of the mounted fiber. Such a calibration was not made during the present study.



Fiber Power Dissipation: 50mW/div

Fiber Resistance: 100 Ω/div

Fiber Voltage Drop: 5V/div

Fiber Current: 5mA/div

0 Reference: -4 division from display center for voltage and current time
-11 division for resistance trace
-12 division for power trace

Figure 22. Carbon Fiber Pulse Heating Parameters - Expanded

Table 4 below summarizes the pulse heating and optical/thermal generation data for a 20 mm mounted fiber.

TABLE 4. PULSE HEATING OF 20 MM CARBON FIBER

Voltage across Fiber (Volts)[1]	10.4	15.0	17.5	20.0	22.5	24.0	25.0	26.0	27.0	28.0	29.0
Fiber Current (m Amps)	6.4	10.6	13.1	16.3	19.5	21.6	22.9	24.8	26.6	28.8	(3)
Power into Fiber (m Watts)	64	159	229	326	439	518	572	645	718	806	
Optical/Thermal Signal (μ Volts)[2]	0.065	0.247	0.714	2.05	5.01	8.05	10.6	14.9	21.3	29.6	

(1) All data taken at 45 msec. into 50 msec. pulse.

(2) Output Voltage of detector angled 45° to fiber with axis of detector intersecting mid-point of fiber and detector face 1 cm removed from that intersection.

(3) Fiber fractured - typical stress fracture positioned in center third of fiber.

These data are plotted in Figure 23 where the log-log scale yields a straight line with a slope of 2.36.

Microwave Fiber Heating--

Figure 24 provides a block diagram of the apparatus used to study the optical/thermal emission from the fiber produced in response to excitation by a microwave field.

A waveguide transmission line and calorimeter load were used so that the microwave field intensity could be precisely determined. In a practical instrument the waveguide would be replaced by a compact cavity excited by a low power microwave source.

Figure 25 shows the mounting of the carbon fiber with respect to the waveguide and the optical/thermal detector. Access to the waveguide interior was provided by a centrally located slot in an H-plane wall. Indexed along this slot was a brass holder which positioned the optical/thermal detector and two 0.010" diamter quartz rods to which the diagonally mounted fiber was attached.

A series of small holes drilled into the brass fixture allowed the quartz rods to be spaced in accordance with the desired carbon fiber length. A small drop of fastsetting epoxy was then placed on each quartz rod at the point where the diagonally positioned carbon fiber would rest. A long fiber was then placed across the quartz rods so as to pass through the epoxy drops. These were allowed to set up and then the excess carbon fiber was trimmed off. This left the carbon fiber stretched at a 45° angle between the quartz rods and held in place by the small epoxy sphere at each end.

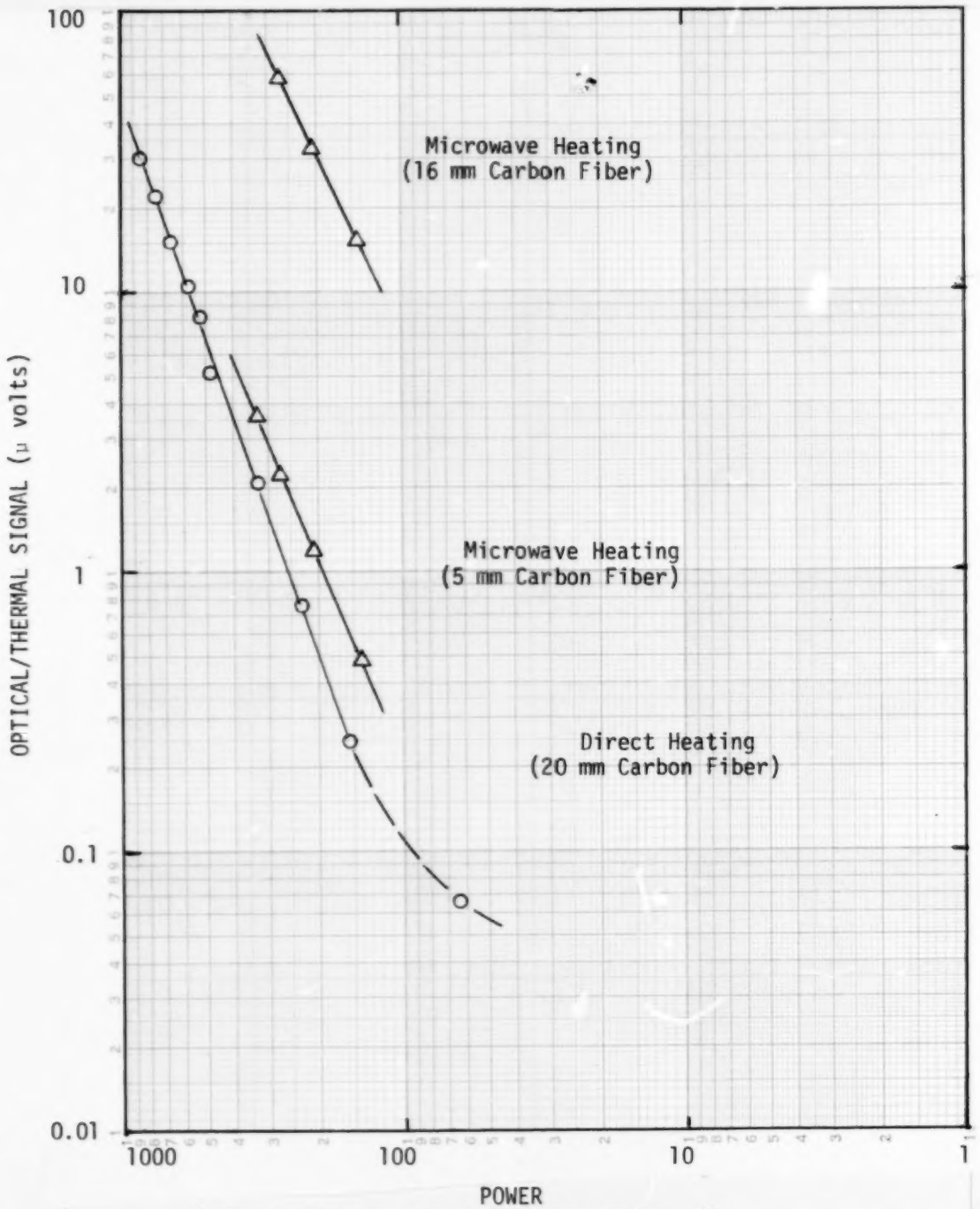


Figure 23. Relationship Between Heating Power and Thermal/Optical Signal for Several Carbon Fiber Lengths

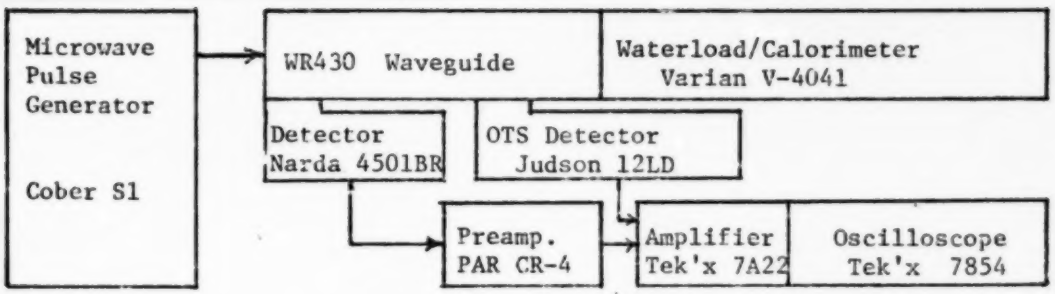


Figure 24. Microwave Excitation Experiment

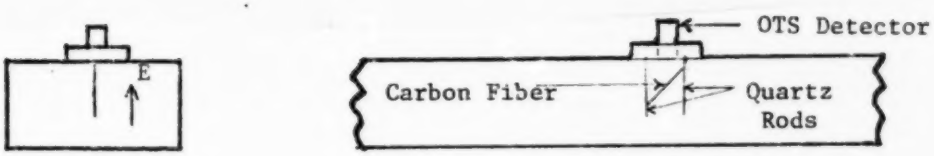


Figure 25. Fiber Mounted in Waveguide

The waveguide E-field reaches a maximum in the plane of the carbon fiber/quartz fiber mounting structure and this maximum intensity is related to the waveguide power by the equation

$$P = 1/4\ (ab)\ Em^2 \sqrt{\varepsilon_o/\mu_o}\ \sqrt{1 - (f_c/f)^2} \qquad (1)$$

where

a = waveguide top-wall inside dimension
b = waveguide side-wall inside dimension

$\varepsilon_o/\mu_o = 376.6\Omega$

f_c = cut-off frequency

f = operating frequency

For WR430 waveguide operating at 2450 MHz

$P = 0.0328\ E^2\ cm^2\ \Omega^{-1}$

It should be noted that this relation holds everywhere in the median E-plane of the waveguide only so long as there are no standing waves. For this reason, special care was taken to obtain a well-matched load, i.e., the Varian V-4041, which has a VSWR < 1.04 at 2450 MHz, and also serves as a calorimeter.

Mounting of the carbon fiber at 45^o to the E-field provides an orientation representative of the average inclination which would be present in a large group of tumbling fibers. Actually, in a monitoring instrument with a sampled flow, the fibers will most probably be preferentially aligned with the flow. Since the E-field inclination should be less than 45^o, an even tighter coupling to the E-field will be achieved than in the present experiment.

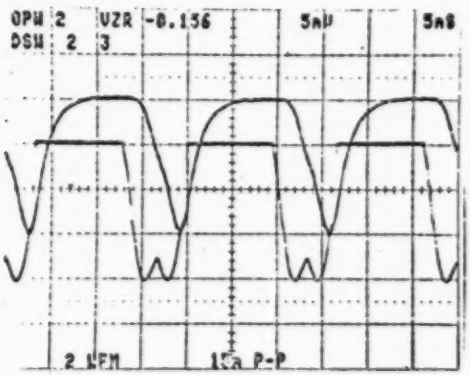
The 45^o carbon fiber mounting also allows the optical/thermal detector to be placed flush with the waveguide wall. No microwave interference with the operation of the detector was found, although the detector did have to be d.c. isolated from the waveguide to prevent ground-loop noise from entering the pre-amplifier. The detector, pre-amplifier, and waveform processing oscilloscope were all operated under the same conditions as in the signal calibration experiment.

The microwave power source for the present experiment was a Cober S1 industrial heating generator which operates at 2450 MHz. The S1 is step-adjustable in power from about 75 watts to about 1000 watts and produces a pulsed output with a 60 Hz repetition rate. The pulsed output is a consequence of the simple, unfiltered, voltage multiplier type of power supply used in the S1 and no adjustment of pulse width or repetition rate is possible.

Monitoring of the pulse waveform was accomplished through the use of a Narda 4501BR envelope detector preceded by a 20 dB attentuator. Sampling of the microwave pulse was by means of a short probe projected into the same waveguide slot used for carbon fiber insertion. As with the optical/thermal detector, d.c. isolation of the sampling probe and envelope detector from the waveguide was necessary to prevent the production of ground loop noise.

Figure 26 shows the typical waveforms obtained from the optical/thermal detector and the microwave pulse envelope detector during excitation of a carbon fiber.

Note that the optical/thermal waveform appears to be delayed with respect to the microwave pulse envelope and that it is quite peaked rather than following the shape of the excitation signal. Both waveform characteristics are the result of a carbon fiber optical/thermal emission rise-time (19 msec.) which is long compared to the width (6 msec.) of the microwave excitation pulse. Since the rise-time of the optical/thermal signal is much less than the width of the microwave pulse, the former never reaches a steady-state value before the latter ends. With a longer microwave pulse, much greater optical/thermal signal amplitudes could have been obtained. However, even the 6 msec. wide microwave pulses provided good carbon fiber signatures.



Optical/Thermal Waveform

Microwave Pulse Envelope

Figure 26. Typical Microwave Envelope and Optical/Thermal Waveforms

Table 5 summarizes the data obtained from the microwave excitation of two representative carbon fibers, one 5 mm in length and the other 16 mm in length. The optical/thermal signals recorded are the peak output values of the indium arsenide detector operating into the low-Z input of the PAR CR-4 preamplifier. The microwave power values given are the average WR-430 waveguide power levels measured by the Varian V-4041 water load/calorimeter. The E-field values are the peak E-fields reached in the mid-plane of the waveguide during the microwave pulse. The actual field experienced by the carbon fiber is the peak field divided by the $\sqrt{2}$, since the fiber is inclined 45^{o} to the E-field.

TABLE 5. CARBON FIBER MICROWAVE HEATING

P^{*} average waveguide (Watts)	E^{*} peak waveguide (Volts/cm)	Peak Optical/Thermal Signal (Volts X 10^{-6})	
		5 mm Fiber	16 mm Fiber
140	109	0.471	15.0
205	132	1.14	32.3
270	151	2.21	57.6
330	167	3.56	fiber fractured

* Duty cycle = 0.36

The above data are plotted in Figure 23 which also contains the optical/thermal signal data for direct pulse heating of the carbon fiber. The slopes for the 5 mm and 16 mm carbon fibers are 2.22 and 2.06 respectively.

Three straight lines connecting the carbon fiber length/optical thermal signal points for the three common waveguide average power levels are plotted in Figure 27. These lines have a slope of approximately 3. Thus, the optical thermal signal has the following approximate dependence on microwave power, P_m, and carbon fiber length, L_c.

$$\text{Optical/Thermal Signal} \propto P_m^2 (L_c)^3$$

An estimate of the power dissipated in the carbon fiber during the microwave pulse can be obtained by matching the optical/thermal signals resulting from microwave heating and from direct pulse heating, provided the former is corrected for the carbon fiber time constant effect, i.e., an optical/thermal signal value is estimated for microwave carbon fiber heating with the same 45 msec. pulse length used in the direct heating experiments.

By definition, the rise time of the optical/thermal signal is the time between the 10% and 90% amplitude values. Since the optical/thermal signal trace seems to reasonably well follow a $1-e^{-\alpha t}$ relationship, the rise time, T_R, and the optical/thermal time constant, α, may be related by

$$\alpha = (\ln 9)/T_R = 0.116 \text{ msec}$$

The ratio of the optical/thermal signal at 45 msec. to that at 6 msec., the signal value recorded, is then given by

$$\text{OTS ratio} = (1-e^{-45\alpha})/(1-e^{-6\alpha}) = 1.98$$

If this correction factor is, for example, applied to the 16 mm carbon fiber in the 151 Volt/cm peak microwave E-field, the resulting optical/thermal signal estimate for 45 msec. microwave excitation is

$$OTS_{45} = \left[(1.98)\ (57.6)\right] = 115 \times 10^{-6} \text{ volts}$$

Comparison with the direct heating curve would indicate the carbon fiber dissipation to be about 1.45 watts, an estimate that is certain to be on the high side, since microwave excitation results in a non-uniform fiber current with greatly increased heating along the center third of the carbon fiber. This increases the optical/thermal emission for a given power input to the fiber.

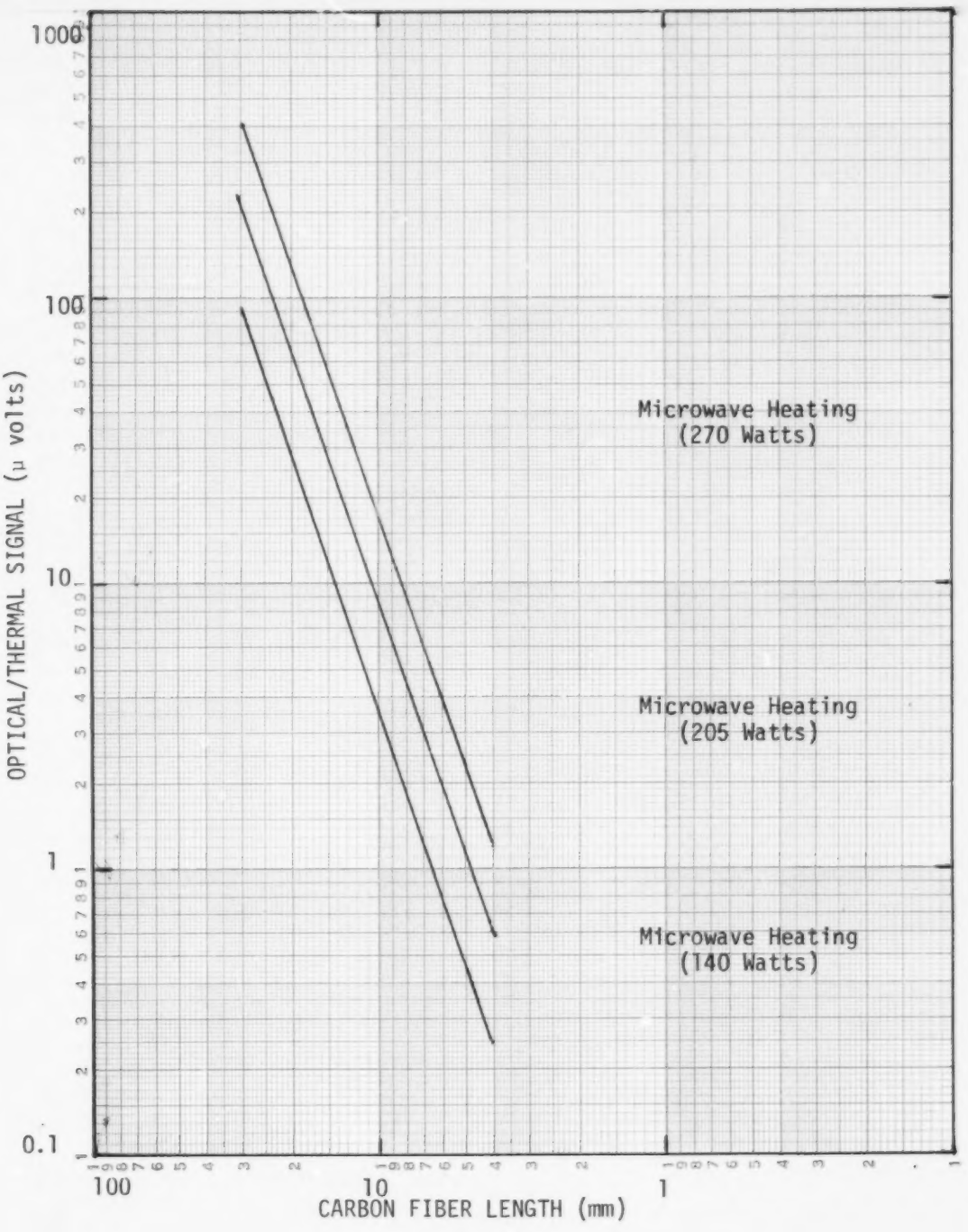


Figure 27. Relationship Between Carbon Fiber Length and Thermal/Optical Signal for Several Common Waveguide Power Levels

EVALUATION CRITERIA

Various instrumental concepts/devices that have been utilized or that have potential for carbon fiber measurement were reviewed in the previous sections. As noted, several of these devices are in the conceptual stage only. Some devices, which are commercially available, are applicable to the selective measurement of fibers of any type while other devices permit measurement of total particulate matter which includes fibers as a fraction of the total.

Each device/concept can be evaluated on the basis of several performance criteria which are indicative of their usefulness for measurement of carbon fibers. These criteria include several factors which define (1) the analytical adequacy of the instrument, (2) the appropriateness of the device to continuously monitor carbon fibers in a manufacturing environment, (3) the physical practicality of the device and (4) the requirement for improvements to achieve adequate operation. Specific evaluation parameters are documented in Table 6. Each parameter has been assigned a weighting factor (WF) on the basis of its perceived importance to carbon fiber measurement.

Carbon fiber concentrations may vary markedly depending on intermittent manufacturing processes occurring at a facility. The ability to measure the concentration of carbon fibers over a wide range is therefore very important. The working concentration range, that is from the detection limit to the saturation point, should be as large as possible to facilitate easiest measurement. The desirable detection limit and saturation point are, of course, a function of the nature of the environment to be sampled.

The size and shape of the particles detected by these instruments are among the most critical factors to be evaluated. The ability to detect carbon fibers at the exclusion of all other particles requires that certain size and aspect ratio values be met. Specifically, carbon fibers have lengths ranging from 5 μm to >10 mm and diameters from <1 to 10 μm. The aspect ratio for such fibers may range from 3:1 to 1000:1. It appears that fibers of 1 to 10 mm in length are most hazardous to electrical equipment while smaller fibers are most hazardous to the human respiratory system.

Measurement Parameters

Within the measurement group, the operating scheme may consist of three alternatives. First, a sample may be extracted from the effluent stream and measured in a separate location. This scheme requires equipment (e.g., pumps) to extract a sample and may alter the condition of the sample between effluent duct and measurement point. Second, a device may be installed in the sample line and measure the concentration of carbon fibers as they flow by. This scheme may require periodic cleaning of the sensing device to maintain accuracy. Third, the carbon fibers may be monitored remotely, thus precluding any interaction of the instrument

TABLE 6. EVALUATION CRITERIA

DETECTION RANGE

Fiber Concentration (fibers/cm^3): 10^{-2} | 1 | 10^2 | 10^4

Fiber Length (cm): 10^{-3} | 10^{-2} | 10^{-1} | 1 | 10

Fiber Aspect Ratio: 3 | 5 | 10 | 20 | 50 | 100

→ Increasing Usefulness ←

EVALUATION PARAMETERS	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1	EXTRACT →	IN-LINE →			REMOTE	
Volume	2	SMALL FRACTION →				LARGE FRACTION	
Time	1	LONG →		SHORT →		CONTINUOUS	
2. DETECTION							
Selectivity	3	PARTICLES-		FIBERS		-------CF	
Basis	2	MASS------		NUMBER		-------BOTH	
Morphology	1	CLUMPS----		BUNDLES----		SINGLES	
Sensitivity	2	LOW				----------HIGH	
Need for fiber orientation	1	YES-----BY METHOD				--------NO	
Detection time	1	MIN.-------SEC.				--------CONT.	
Frequency of error	2	OFTEN				----------SELDOM	
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1	LARGE				----------SMALL	
Power requirements	1	HIGH				----------LOW	
Ruggedness	2	FRAGILE				----------RUGGED	
Maintenance	1	OFTEN				----------SELDOM	
Calibration	1	NONE				----------PRIMARY	
Unit cost	2	HIGH				---------- LOW	
Development cost	1	HIGH				----------LOW	

4. DISADVANTAGES (Negative Scores)

Concept only - no experimental verification	(-10)
Based on limited laboratory experiments	(-5)
Other:	( )
i.e., High false positive count in moist stream	(-3)
Concentration if function of velocity	(-3)

Sub Total ☐

LESS ☐

TOTAL SCORE ☐

with a corrosive medium. This latter scheme is ranked as most useful although it is acknowledged that it may be the least suitable on other grounds.

The sample volume is of importance for two reasons. A larger sample potentially provides a lower detection level and a more representative sample. A sampling interval of small duration is ultimately preferred since it provides maximum protection against short term release of environmentally hazardous fibers. Continuous monitoring provides such protection and can yield time-weighted averages of fiber concentration.

Detection Parameters

Selectivity and sensitivity are of critical importance for environments containing carbon fibers, non-conductive fibers and other particles. This may not be necessary in some environments. As noted above, the morphology of carbon fibers, i.e., clumps, bundles, single fibers, is of importance due to their aerodynamic behavior. Single fibers are generally of most concern. One other factor affecting detection in many cases is the need for a specific fiber orientation. As an example, a fiber may pass undetected through an electric grid if it is oriented perpendicular to the plane of the grid or a carbon fiber may not be differentiated from a non-conductive fiber in a light scattering device if it does not rotate in the sensing field. It is apparent that if fibers need to be oriented, more complex instrumentation is required. The simplest devices do not require orientation.

The frequency of failure of the instrument to detect a carbon fiber or the frequency of false alarm detection is important in terms of hazardous protection and cost of system operation, respectively. It is quite obvious that fewer errors are more desirable.

Instrument Parameters

Instrument parameters included in Table 6 are generally self-explanatory. Some parameters, such as size and power requirements, are dependent on sampling environments. In other cases, (e.g., ruggedness; unit cost) the parameter relates to the usefulness of the device.

Other Considerations

Also included in the evaluation criteria are a subjective ranking of deficiencies or areas where improvements are required to increase the sensitivity or selectivity of these methods or concepts for carbon fibers.

Ranking of Methods

Weighing factors for individual parameters were chosen on the expectation that detection parameters are of most importance followed by measurement parameters and instrument parameters in that order. However, since the weighting factor is critically related to the needs in a specific

sampling environment final ranking (based on numerically-weighted scores) of all methods is given on the basis of three sampling scenarios described previously.

COMPARISON OF METHODS

Evaluation sheets were prepared for each of the candidate methods listed in Table 2, following the model presented in Table 6. These evaluation sheets are presented in Appendix A. With consideration to the estimated fiber concentration (number) and length ranges for these methods, it is noted that the methods tend to fall into one of three regions, as follows:

1. Very high concentration, long length

   - Optical-opacity
   - Microwave interception
   - Radar
   - Infrared

2. Moderate concentration, long length

   - Ball gauge
   - Electric grid - arc
   - Electric grid - resistance
   - Microwave - OSGEF

3. Moderate concentration, short length

   - Optical scattering

These regions, which do not overlap, are shown in Figure 28 together with the location of the three emission scenarios given in Table 1. Scenarios A and C fall near boundries of the Region 2 methods. Scenario B would be detected by most of the Region 1 methods; however, these methods measure total particulate and are not specific to carbon fibers thereby limiting their usefulness to specific applications. It should be pointed out that the Region 2 methods would detect the presence of Scenario B carbon fibers but would underestimate their concentration. Such a device could still be useful for indicating an excursion in carbon fiber concentration above some acceptable level.

The Region 3 optical scattering methods are expected to cover a wide range of concentration but are limited to relatively short fibers. These methods would be the only useful approach for monitoring respirable fiber sizes and could be used in conjunction with one of the Region 2 methods for complete coverage of any length of carbon fiber over three to four decades of concentration.

Methods corresponding to each of the length-concentration regions and their evaluation scores are as follows:

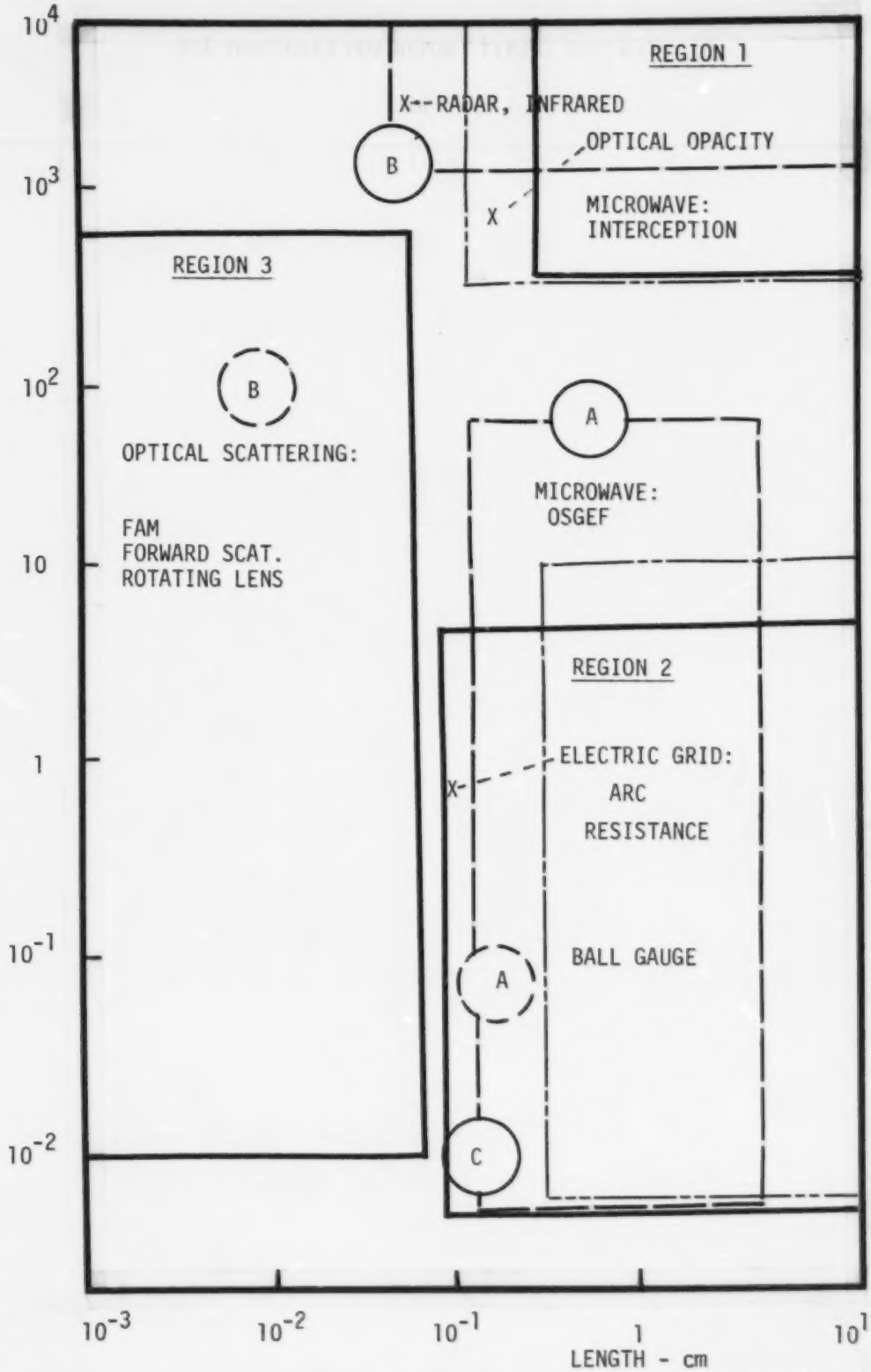


Figure 28. Detection Range of Candidate Carbon Fiber Monitor Methods (See Figure 1 for description of Scenarios A,B and C)

No.	Method	Score
	Region 1 (High Concentration, Long Length)	
4	Optical - Opacity	52
8	Microwave - Interception	56
10	Remote - Radar	56
11	Remote - Infrared	56
	Region 2 (Moderate Concentration, Long Length)	
1	Ball Gauge	77
2	Electric Grid - Arc	79
3	Electric Grid - Resistance	75
9	Microwave - OSGEF	79
	Region 3 (Moderate Concentration, Short Length)	
	Optical Scattering:	
5	Fiber Aerosol Monitor	69
6	Near Forward Scattering	60
7	Rotating Lens	61

The methods which are felt to be most promising for application as a carbon fiber monitor are as follows:

1. Fibers 1 mm and longer in presence of background particulate.

   - Microwave OSGEF - The major advantage of the method is the highly specific identification of carbon fibers in the presence of other matter, including conductive particles and fibers. The method should be applicable over a large dynamic range of fiber concentration. Further development studies are required to establish detection limits with respect to measurement time, flow rate (volume), minimum length and maximum concentration, the latter being influenced mainly by particle coincidence. The detection section of the instrument is simple, and data processing and display can be remote.

   - Electric Grid - Arc - The device may be constructed to intercept a major portion of a flowing airstream, thereby providing good representativeness. Detection is based on particle interception between two or more electrodes. Fiber counts can be obtained over a large dynamic range. The method is only approximately length specific, depending on electrode spacing, and fibers suspended parallel to the airstream may be missed. False positive counts are possible depending on the nature (conductivity, size) of other particulate matter. Data processing and display can be remote. Data processing for length and

and mass concentration may be complex. The instrument may require frequent cleaning and calibration, depending on the nature of other particulate and entrained moisture.

2. Very high concentration of fibers in absence of other particulate matter.

   - Optical-Opacity - The light-emitting diode (LED) is simple and inexpensive; it is suitable for detecting a very high emission of carbon fibers in the absence of other particulate matter. For example, this device could trigger an alarm in the event of a process upset where a very large number of fibers were emitted and drawn through an exhaust hood duct. The device is simple and inexpensive, but is limited to a measure of total particulate matter, giving no information about carbon fiber size, number or mass concentration.

3. Respirable Fibers

   - Optical Scattering - Fiber Aerosol Monitor - The usefulness of the FAM instrument has been demonstrated for the measurement of asbestos fibers. With some modifications to increase sampling volume and the length of fibers detected, the instrument could be usefully applied to measure the number and size distribution of carbon fibers. The instrument is complex, expensive and cannot distinguish between fiber type. Based on results for measuring asbestos, the measurement is accurate and precise.

   - Optical Scattering - Rotating Lens - This method is based on a concept. Further design and laboratory evaluation is required. The method offers the possibility for measuring a large range of fiber lengths, limited only by particle (or fiber) coincidence. The "sampling" of the stream is remote and can be arranged to traverse across the duct. As with other optical scattering methods, fibers of all types are measured.

SECTION 7

PHASE II DEVELOPMENT PROGRAM

Based on a review of possible sources for carbon fiber emissions during manufacturing, processing or disposal, and on an evaluation of methods or candidates for continuously monitoring carbon fiber emissions in ducts or stacks, it has been determined that no single instrument or concept is available for measuring the entire range of concentrations and fiber lengths that may be encountered. The EPA must determine the relative importance of several parameters and conditions, such as those raised by the following questions:

- Is an alarm showing that a preset carbon fiber concentration (or mass) has been exceeded acceptable or is a continuous readout of concentration needed?
- What ranges of fiber length are most important? How significant is a specification of length distribution?
- What are restrictions or requirements relative to unit cost, instrument calibration, representativeness of sampling, etc.

Without more specific guidelines regarding these issues, only general guidance can be provided concerning recommendations for a Phase II development program.

As described in the previous section, five carbon fiber monitoring methods or concepts are selected as worthy of further consideration, depending on the specific needs of the EPA. Recommendations for development programs and associated provisos are given in Table 7.

The microwave-OSGEF method is felt to be the superior method for carbon fiber monitoring, being the only method that is entirely carbon fiber specific. Optical/thermal signals measured in the current laboratory study represent a worst case, with an expected increase of 2 to 3 times for more optimum heating conditions. Further, the measurements were carried out on a carbonized fiber (only partially graphitized) whereas market projections indicate that the demand for graphitized fibers is increasing more rapidly. A fully graphitized fiber, having a positive temperature coefficient of resistance should achieve higher temperatures and more rapidly than for carbonized fibers, further increasing signal. The method requires extractive sampling with a nozzle, pump and flow meter, thereby being more complex than a sensor placed in the stack or duct.

The electric grid-arc method should also be developed for carbon fiber monitoring. Although less specific to carbon fibers and subject to false positives, the method is simple and can cover a larger cross-sectional area of the duct than other methods, providing better representation of the stream. It is unlikely that the method can provide as good a description of length distribution as OSGEF. Experimental programs are currently underway which should provide better definition of the advantages and shortcomings of the method.

The other three methods listed in Table 6 represent special cases. The optical scattering-rotating lens is at the concept stage and represents a higher risk of success than other methods, but with a higher payoff in terms of larger dynamic range of fiber lengths that can be detected and remote sensing. A laboratory-based concept demonstration program is recommended. The optical LED method is only appropriate in selected applications as an alarm to indicate high levels of particulate matter emissions. The instrument is available and inexpensive. The cost of a field evaluation program to determine its suitability for signaling the release of carbon fibers as a result of process upsets would be relatively low.

Finally, if it is established that there is a need to measure carbon fibers of respirable size range, modification to the Fiber Aerosol Monitor developed by GCA Corp. and generally used for monitoring asbestos fiber concentrations is recommended. The instrument measures all fibers, which is a drawback for applications in which both carbon fibers and glass fibers are present. The major advantage of this instrument is the level of development as an asbestos monitor, with obvious tradeoffs in the already developed procedures for sample extraction, calibration and signal processing.

TABLE 7. RECOMMENDED PHASE II DEVELOPMENT PROGRAMS

PRIORITY	METHOD	APPLICATION		REQUIREMENT*				ESTIMATED COST* ($1000's)			
		Length (mm)	Concentration (fibers/cm^3)	A	B	C	D	A	B	C	D
1	Microwave-OSGEF+	1-50	10^{-2} - 10^{2}			X	X			120-140	20-25
1	Electric grid-arc	1-100	10^{-2} - 10			X†	X			(?)	15-20
2	Optical-rotating# lens	10^{-3}-1	10^{-2} - 10^{2}	X**	X			5	30-50		
3	Optical-LED	10^{-3}-1	10^{-2}-10^{2}				X				10-20
3	Optical-FAM++	--	>10^{3}			X	X			(?)	20-25

* A - Develop theory and preliminary design.
B - Conduct laboratory demonstration of concept.
C - Build prototype and test in laboratory.
D - Modify prototype and field test.

\+ This concept was developed by E. Cook, Arthur D. Little, Inc., Cambridge, MA 02140.

† Current programs with Bionetics and JPL should satisfy this requirement.

\# This concept was developed by H. Miranda, Epsilon Laboratories, Inc., Bedford, MA.

**Much of the theoretical development and design was previously carried out for a proposal to RADC; no program was funded under this RFP.

++The FAM is sold by GCA Corp., Bedford, MA 01730.

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TOP

APPENDIX A

CANDIDATE METHOD EVALUATION SHEETS

Sheet No.	Method
1	Ball Gauge
2	Electric Grid - Arc
3	Electric Grid - Resistance
4	Optical - Opacity
5	Optical - Scattering: Fiber Aerosol Monitor
6	Optical - Scattering: Near Forward Scattering
7	Optical - Scattering: Rotating Lens
8	Microwave - Interception
9	Microwave: OSGEF
10	Remote: Radar
11	Remote: Infrared

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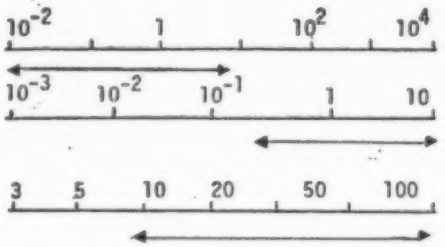
EVALUATION SHEET NO. 1

METHOD Ball Gauge

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2		x				4
Time	1				x		4
2. DETECTION							
Selectivity	3			x			9
Basis	2			x			6
Morphology	1				x		4
Sensitivity	2				x		8
Need for fiber orientation	1				x		4
Detection time	1				x		4
Frequency of error	2			x			6
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1			x			3
Ruggedness	2				x		8
Maintenance	1		x				2
Calibration	1		x				2
Unit cost	2			x			6
Development cost	1				x		4

4. DISADVANTAGES (Negative Scores) 80

Concept only - no experimental verification ( )
Based on limited laboratory experiments ( )
Other: Count influenced by distance from ball, mass and velocity ( )
(-3)

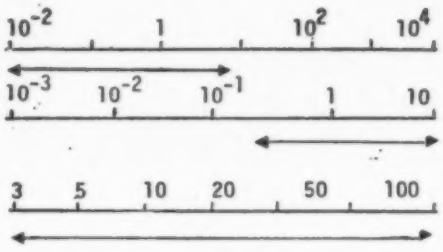
-3

TOTAL SCORE 77

EVALUATION SHEET NO. 2

METHOD Electric Grid - Arc

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3			x			9
Basis	2				x		8
Morphology	1				x		4
Sensitivity	2			x			6
Need for fiber orientation	1			x	x		3
Detection time	1				x		4
Frequency of error	2			x			6
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1				x		4
Power requirements	1		x				2
Ruggedness	2			x			6
Maintenance	1		x				2
Calibration	1		x				2
Unit cost	2				x		8
Development cost	1				x		4
							82

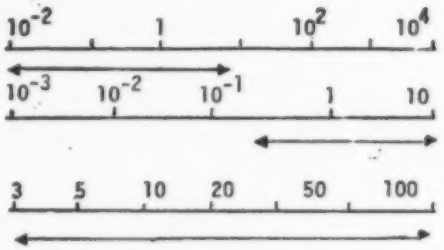
4. DISADVANTAGES

Concept only - no experimental verification	( )
Based on limited laboratory experiments	( )
Other:	( )
Can have high false positive count due to moisture on particles, etc.	(-3)
	-3
TOTAL SCORE	79

EVALUATION SHEET NO. 3

METHOD Electric Grid - Resistance

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3			x			9
Basis	2				x		8
Morphology	1				x		4
Sensitivity	2			x			6
Need for fiber orientation	1				x		4
Detection time	1			x			3
Frequency of error	2			x			6
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1		x				2
Ruggedness	2			x			6
Maintenance	1		x				2
Calibration	1		x				2
Unit-cost	2			x			6
Development cost	1			x			3
							78

4. DISADVANTAGES

Concept only - no experimental verification	( )
Based on limited laboratory experiments	( )
Other:	( )
Can have high false positive count due to moisture on particle, etc.	(-3)

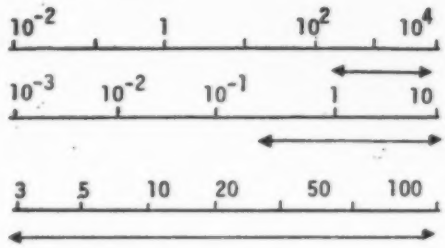
-3

TOTAL SCORE 75

EVALUATION SHEET NO. 4

METHOD Optical - Opacity

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3	x					3
Basis	2	x					2
Morphology	1	x					1
Sensitivity	2	x					2
Need for fiber orientation	1					x	5
Detection time	1					x	5
Frequency of error	2	x					2
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1	x					1
Power requirements	1		x				2
Ruggedness	2		x				4
Maintenance	1				x		4
Calibration	1			x			3
Unit cost	2		x				4
Development cost	1				x		4
							55

4. DISADVANTAGES

Concept only - no experimental verification	( )
Based on limited laboratory experiments	( )
Other:	( )
Limited to high concentrations and absence of other particulate	(-3)

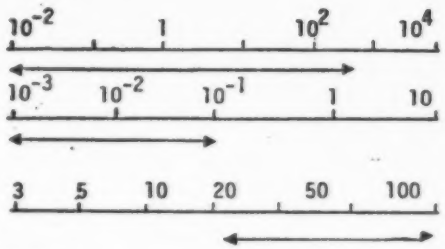
-3

TOTAL SCORE 52

EVALUATION SHEET NO. 5

METHOD Optical-Scattering: Fiber Aerosol Monitor

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1	x					1
Volume	2	x					2
Time	1				x		4
2. DETECTION							
Selectivity	3		x				6
Basis	2				x		8
Morphology	1			x			3
Sensitivity	2				x		8
Need for fiber orientation	1			x			3
Detection time	1				x		4
Frequency of error	2				x		8
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1			x			3
Ruggedness	2		x				4
Maintenance	1		x				2
Calibration	1				x		4
Unit-cost	2		x				4
Development cost	1				x		4
							71

4. DISADVANTAGES

Concept only - no experimental verification ( )
Based on limited laboratory experiments ( )
Other: ( )
Not suitable for detecting clumps or bundles (-2)

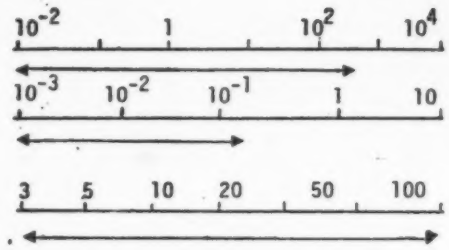
-2

TOTAL SCORE 69

EVALUATION SHEET NO. 6

METHOD Optical Scattering: Near Forward Scattering

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1	x					1
Volume	2	x					2
Time	1	x			x		4
2. DETECTION							
Selectivity	3		x				6
Basis	2			x			6
Morphology	1			x			3
Sensitivity	2				x		8
Need for fiber orientation	1				x		4
Detection time	1				x		4
Frequency of error	2			x			6
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1			x			3
Ruggedness	2		x				4
Maintenance	1		x				2
Calibration	1			x			3
Unit-cost	2		x				4
Development cost	1		x				2
							65

4. DISADVANTAGES

Concept only - no experimental verification ( )
Based on limited laboratory experiments (-5)
Other: ( )
( )
( )
( )

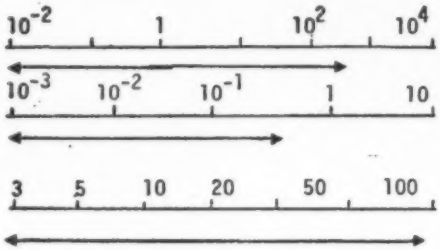
-5

TOTAL SCORE 60

EVALUATION SHEET NO. 7

METHOD Optical Scattering: Rotating Lens

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2	x					2
Time	1				x		4
2. DETECTION							
Selectivity	3		x				6
Basis	2				x		8
Morphology	1				x		4
Sensitivity	2				x		8
Need for fiber orientation	1					x	5
Detection time	1				x		4
Frequency of error	2			x			6
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1		x				2
Power requirements	1			x			3
Ruggedness	2		x				4
Maintenance	1			x			3
Calibration	1			x			3
Unit cost	2		x				4
Development cost	1		x				2
							71

4. DISADVANTAGES

Concept only - no experimental verification (-10)
Based on limited laboratory experiments ( )
Other: ( )
( )
( )
( )

-10

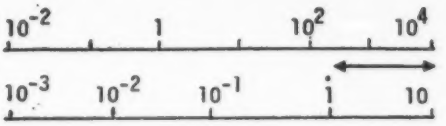
TOTAL SCORE 61

EVALUATION SHEET NO. 8

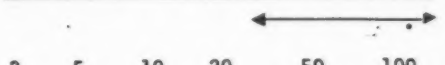
METHOD Microwave: Interception

DETECTION RANGE

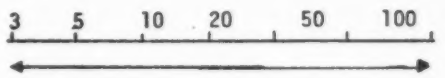
Fiber Concentration (fibers/cm^3)



Fiber Length (cm)



Fiber Aspect Ratio



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3	x					3
Basis	2	x					2
Morphology	1	x					1
Sensitivity	2	x					2
Need for fiber orientation	1					x	5
Detection time	1					x	5
Frequency of error	2	x					2
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1			x			3
Ruggedness	2			x			6
Maintenance	1			x			3
Calibration	1			x			3
Unit cost	2		x				4
Development cost	1			x			3
							59

4. DISADVANTAGES

Concept only - no experimental verification	( )
Based on limited laboratory experiments	( )
Other:	( )
	( )
Limited to high concentration and absence of other particulate	(-3)
	( )

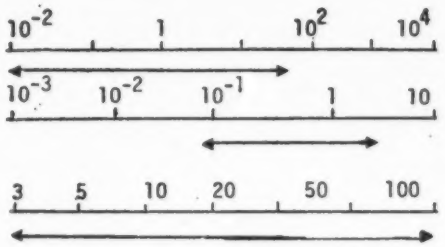
-3

TOTAL SCORE 56

EVALUATION SHEET NO. 9

METHOD Microwave: OSGEF

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1			x			3
Volume	2	x					2
Time	1				x		4
2. DETECTION							
Selectivity	3					x	15
Basis	2				x		8
Morphology	1				x		4
Sensitivity	2				x		8
Need for fiber orientation	1					x	5
Detection time	1				x		4
Frequency of error	2				x		8
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1			x			3
Power requirements	1			x			3
Ruggedness	2			x			6
Maintenance	1			x			3
Calibration	1		x				2
Unit cost	2		x				4
Development cost	1		x				2

4. DISADVANTAGES

Concept only - no experimental verification ( )
Based on limited laboratory experiments (-5)
Other: ( )
( )
( )
( )

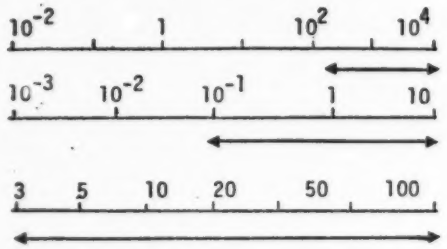
-5

TOTAL SCORE 79

EVALUATION SHEET NO. 10

METHOD Remote Sensing: Radar

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1					x	5
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3		x				6
Basis	2	x					2
Morphology	1	x					1
Sensitivity	2	x					2
Need for fiber orientation	1					x	5
Detection time	1					x	5
Frequency of error	2	x					2
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1	x					1
Power requirements	1		x				2
Ruggedness	2				x		8
Maintenance	1			x			3
Calibration	1	x					1
Unit cost	2	x					2
Development cost	1			x			3
							59

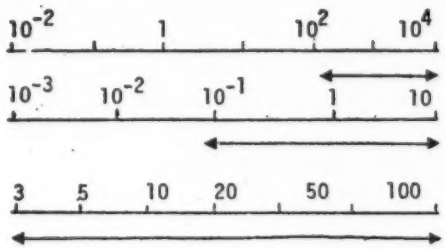
4. DISADVANTAGES

Concept only - no experimental verification	(-10)	
Based on limited laboratory experiments	(- 5)	
Other:	( )	
	( )	
Limited to high concentrations and absence of other particulate	(-3)	
	( )	
	( )	-3
TOTAL SCORE		56

EVALUATION SHEET NO. 11

METHOD Remote Sensing: Infrared

DETECTION RANGE



PARAMETER	WF	VALUE 1	2	3	4	5	SCORE
1. MEASUREMENT METHOD							
Sampling	1					x	5
Volume	2			x			6
Time	1					x	5
2. DETECTION							
Selectivity	3		x				6
Basis	2	x					2
Morphology	1	x					1
Sensitivity	2	x					2
Need for fiber orientation	1					x	5
Detection time	1					x	5
Frequency of error	2	x					2
3. INSTRUMENT PARAMETERS							
Physical size (weight)	1	x					1
Power requirements	1		x				2
Ruggedness	2				x		8
Maintenance	1			x			3
Calibration	1	x					1
Unit-cost	2	x					2
Development cost	1			x			3

4. DISADVANTAGES

Concept only - no experimental verification	( )	
Based on limited laboratory experiments	( )	
Other:	( )	
Limited to high concentrations and absence of other particulate	(-3)	-3
	( )	
	( )	
TOTAL SCORE		56

59